Attorney Docket No. 1021238-000578

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Patent Application of	
Rajesh K. GARG et al.	Group Art Unit: 1731
Application No.: 10/649,787	Examiner: Jose A. FORTUNA
Filed: August 28, 2003	Appeal No.: Unassigned
For: METHOD AND APPARATUS FOR PREPARING A SLURRY OF ADD-ON MATERIAL TO BE APPLIED TO A WEB	

APPEAL BRIEF

Mail Stop APPEAL BRIEF - PATENTS
Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Sir:

This appeal is from the decision of the Primary Examiner dated February 16, 2007 finally rejecting Claims 1-5, which are reproduced as the Claims Appendix of this brief.

☐ A check covering the ☐ $ 250 ☐ $ 500 Government fee is filed herewith.

☒ Charge ☐ $ 250 ☒ $ 500 to Credit Card. Form PTO-2038 is attached.

The Commissioner is hereby authorized to charge any appropriate fees under 37 C.F.R. §§1.16, 1.17, and 1.21 that may be required by this paper, and to credit any overpayment, to Deposit Account No. 02-4800.

I. Real Party in Interest

The above-identified patent application is assigned to PHILIP MORRIS USA INC. PHILIP MORRIS USA INC. is the real party in interest.

II. Related Appeals and Interferences

Neither Appellants' legal representatives, nor the assignee, knows of any other appeal or interference which will affect, or be directly affected by, or have bearing on, the Board's decision in the pending appeal.

III. Status of Claims

Claims 1-22 were originally presented for examination.

Claims 1-5 stand as rejected.

Claims 6-22 have been cancelled.

The final rejection of pending Claims 1-5 is hereby appealed.

IV. Status of Amendments

Following the final rejection, Claim 1 was amended. *See* Amendment Pursuant to 37 C.F.R. § 1.116 filed May 16, 2007. The Advisory Action mailed June 1, 2007, indicated that the proposed amendment to Claim 1, filed after the final rejection, would not be entered. That proposed amendment of Claim 1 is not included in the Appealed Claims reproduced in the Claims Appendix.

V. Summary Claimed Subject Matter

Independent Claim 1 is directed to a method of manufacturing a web having an applied pattern of add-on material[1], the method comprising moving a base web along a first path[2]; preparing a slurry of add-on material[3]; and repetitively discharging said slurry of add-on material upon said moving base web[4], the step of preparing a slurry of add-on material

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[1] See, for example, specification, Page 2, Lines 14-18; Page 5, Lines 7-10 and 21-24; FIG. 1, References 22 and 40; and FIG. 2, References 3 and 5.

[2] See, for example, specification, Page 5, Lines 5-10; Page 5, Lines 9-10; and FIG. 1, Reference 22.

[3] See, for example, specification, Page 2, Lines 12-15, and Page 5, Lines 25-26.

[4] See, for example, specification, Page 5, Lines 5-10; Page 5, Lines 7-10; and FIG. 1, References 22 and 40.

including: cooking a fibrous cellulosic material[5], bleaching the material[6], pressing the cooked and bleached material to remove liquid[7], drying the pressed material[8], milling the dried material to produce fibers of a desired size[9], and mixing the milled material with water to hydrate the material and produce a slurry[10].

Claim 4, which is dependent on Claim 3[11], specifies that the method further includes subjecting the flax straw feed stock to a process for removing non-fibrous component including shive before the step of cooking the fibrous cellulosic material[12].

Claim 5, which is dependent on Claim 4, specifies that the process for removing the non-fibrous component is preformed in a hammer mill[13].

VI. Grounds of Rejection to be Reviewed on Appeal

Claims 1-5 have been finally rejected under 35 U.S.C. §103(a) as unpatentable over U.S. Patent No. 5,997,691 ("Gautam").

VII. Argument

As noted above, Claims 1-5 have been finally rejected under 35 U.S.C. §103(a) as unpatentable over Gautam. The Examiner cites Gautam as teaching:

> a method of making a web in which a base web is moved along a first path, a slurry of cellulosic material is prepared as an[] add-on to the base web; and repetitively discharging the add-on-material. . . . [T]he add-on material is

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[5] See, for example, specification, Page 2, Line 10; Page 5, Line 25 – Page 6, Line 1; Page 6, Lines 7-8; FIG. 3, References 190 and 200; and FIG. 4, Reference B.

[6] See, for example, specification, Page 2, Line 10; Page 6, Lines 7-8; FIG. 3, Reference 210; and FIG. 4, References 122 and B.

[7] See, for example, specification, Page 2, Lines 10-11; Page 6, Lines 9-10; and FIG. 4, Reference 124.

[8] See, for example, specification, Page 2, Line 11; Page 6, Line 10; and FIG. 4, Reference 124.

[9] See, for example, specification, Page 2, Line 12; Page 6, Lines 10-12; and FIG. 4, Reference 126.

[10] See, for example, specification, Page 2, Lines 12-15; Page 6, Lines 22-24; and FIG. 4, Reference 128.

[11] Claim 3 specifies that the fibrous cellulosic material comprises flax straw feed stock; see, for example, specification, Page 4, Lines 19-21; Page 5, Line 25 – Page 6, Line 6; FIG. 3, Reference 190; and FIG. 4.

[12] See, for example, specification, Page 6, Lines 1-6.

[13] See, for example, specification, Page 6, Lines 1-6.

> discharged using a moving belt having an orifice along the endless path. . . . [F]lax straw [is used] as the add-on material. . . . [T]he add-on material is cooked, bleached and then grinded, i.e., refined." (Final Office Action, Page 3).

Admitting that Gautam fails to teach the recited feature of milling the dried material (which the Office Action appears to equate with "dry grinding"), the final Office Action further asserts,

> The only difference between the claimed invention and Gautam et al. invention is that the way in which the add-on material is ground, i.e., Gautam et al. teach a wet grinding process, while the present application teaches the dry comminution [footnote deleted] of the add-on materials. However, using either process of grinding is within the levels of ordinary skill in the art, since both of them are very well known in the art. Note that if one desires to do the dry grinding operation, then the steps of pressing and drying the slurry are a necessary and also very well known in the dry market pulp. Wet and dry grinding are functional equivalent processes. . . . (Final Office Action, Page 3).

Appellant respectfully disagrees with the rejection of Claims 1-5 as unpatentable over Gautam. Therefore, reversal of this rejection is respectfully requested.

A. <u>Claims 1-5</u>

1. <u>Background</u>

In a Response filed November 21, 2006, Appellants asserted that the Office Action mailed July 24, 2006, did not provide any factual basis for its assertion that wet and dry grinding are functionally equivalent processes with regard to the preparation of add-on material, a pattern of which is to be applied across a base width of cigarette paper. Since Appellants challenged a factual assertion of the Office Action as not properly officially noticed or not properly based upon common knowledge, the Examiner was requested to provide documentary evidence in the next Office Action if the rejection was to be maintained.

The final Office Action asserted that the prior Office Action mailed July 24, 2006, cited enough evidence of the equivalence of wet and dry grinding, referring to the references cited in the footnote on page 3. But, in the Amendment filed May 16, 2007, Appellants pointed out that the references cited in the footnote on page 3 were cited "[f]or example of dry grinding operation", rather than the equivalence of wet and dry grinding in paper manufacture. Thus, the final Office Action further cited U.S. Patent No. 3,596,840 ("Blomqvist"), U.S. Patent No. 6,214,166 ("Münchow"), and U.S. Patent Application

Publication No. 2005/0167534 ("Tomikawa") as evidence of the equivalence of wet and dry grinding. Münchow and Tomikawa were cited for the first time in the final rejection.

"In order to rely on a reference as a basis for rejection of an applicant's invention, the reference must either be in the field of applicant's endeavor or, if not, then be reasonably pertinent to the particular problem with which the inventor was concerned." *In re Oetiker*, 977 F.2d 1443, 1446, 24 USPQ2d 1443, 1445 (Fed. Cir. 1992); MPEP § 2141.01(a).

Appellants respectfully submit that none of Blomqvist, Münchow, or Tomikawa provide evidence of the alleged equivalence between wet and dry grinding, either in a papermaking process or in a method for preparing a slurry of add-on material to be applied in a predetermined pattern on a base web. Moreover, none of Blomqvist, Münchow, or Tomikawa is reasonably pertinent to the particular problem with which the Appellants were concerned or in the field of Appellants' endeavor, namely, a method and apparatus for preparing a slurry of add-on material to be applied in a predetermined pattern on a base web, preferably in the form of bands, and more particularly, to a method and apparatus for producing cigarette paper having banded regions of the additional material. (Page 1, Paragraph [0001]). Furthermore, contemporaneous materials confirm that dry grinding is not equivalent to wet grinding in papermaking processes. Accordingly, the Blomqvist, Münchow, and Tomikawa references do not properly support the rejection of Claims 1-5.

2. <u>The Gautam Patent</u>

The Gautam patent is the single reference relied upon to reject Claims 1-5. But Gautam does not disclose any dry grinding process or apparatus. As explained in the present specification, "dry grinding" results in add-on material having a very narrow range of cellulose fiber lengths, and as a result the areas of the cigarette paper having the add-on material provide consistent and predictable performance. In addition, the add-on material is produced in much shorter time while consuming less energy than would be required to produce similar add-on material having a comparably narrow range of fiber lengths using techniques wherein a wet slurry material is repeatedly refined using multi-disk refiners. (Page 3, Paragraph [0010]).

3. The Blomqvist Patent

Blomqvist provides a process for producing cellulose fluff, a disintegrated dry cellulose fiber product used as an absorbent material in such items as diapers, absorbent pads and rolls, and the like. (Abstract). The final Office Action asserted that Blomqvist "teaches in column 1, lines 46-56, some of the advantages of using dry grinding, instead of wet grinding." (Final Office Action, Page 4). However in column 1, Blomqvist in fact teaches some of the advantages of producing cellulose fluff starting from pulp in sheet form, instead of loosely compressed roll pulp. With regard to wet grinding, Blomqvist merely discloses, "it is surprising that it is possible to treat dry pulp in a disk refiner in which otherwise only wet pulp can be treated." (Column 1, Lines 58-60). Moreover, Blomqvist expressly notes that earlier experiments showed that pulp could be burnt in the refiner (Column 1, Lines 60-63) – a fact which precludes use in paper where brightness is one of the most important characteristics.[14] Thus, Blomqvist does not provide any evidence of the equivalence of wet and dry grinding in papermaking. Rather, the Blomqvist process produces dry cellulose fluff fibers as a final product for use as an absorbent material, but not a slurry formed by mixing disintegrated (fluffed) dry cellulose fibers with water to hydrate the disintegrated (fluffed) dry cellulose fibers.

4. The Münchow Patent

Münchow relates to a process for recycling fillers and coating pigments during the preparation of paper, paperboard and cardboard. Those fillers and pigments are found in the residual water sludges from coating-plant waste waters, deinking plants, internal water treatment plants or separators. A pigment slurry obtained from the process is then used to prepare a coating compound for the paper industry or as an additive to paper stock for papermaking. (Abstract). Münchow discloses that the residual water sludges are first given desired whiteness and fineness by mixing and then milling together with (i) fresh pigments, (ii) fresh fillers in the form of powders, (iii) fresh-pigments containing slurries, and/or (iv) fresh-filler containing slurries. That processed sludge is then used as a filler or coating pigment. (Column 3, Lines 17-23). Münchow further discloses, "The mineral fillers and pigments mentioned are usually milled to give the desired grain size in a wet or dry milling

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[14] *See, e.g.*, http://www.paperonweb.com/pulppro.htm.

method." (Column 3, Lines 23-25). Thus, Münchow does not provide any evidence of the alleged equivalence of wet and dry grinding to attain desired fiber length in a papermaking process, specifically of an add-on material comprising fibrous cellulosic material.

5. <u>The Tomikawa Application</u>

Tomikawa also concerns a dry grinding system which is suitable for use in production, for example, of abrasives or filler; as well as a dry grinding method employing the system. (Page 1, Paragraph [0003]). Tomikawa discloses that, in general, ceramic powder such as alumina powder or silicon carbide powder (employed as, for example, abrasive or fillers), is produced through grinding of raw-material powder having a large average particle size. (Page 1, Paragraph [0003]). Tomikawa further states, "Grinding processes include a dry grinding process and a wet grinding process. When a dry product is to be produced by means of a grinding process, in many cases, a dry grinding process, which does not require a drying step, is employed." (Page 1, Paragraph [0003]). Thus, Tomikawa does not provide any evidence of the equivalence of wet and dry grinding in a papermaking process. Tomikawa indicates that raw-material ceramic powder having a large average particle size may be ground to produce ceramic powder (such as alumina powder or silicon carbide powder), where the ceramic powder may then be used, for example, as an abrasive or filler – but does not disclose mixing a milled fibrous cellulosic material with water to hydrate that material and produce a slurry.

6. <u>Wet Grinding and Dry Grinding are Not Equivalent Processes</u>

Appellants' Amendment Pursuant to 37 C.F.R. § 1.116 has not been entered. Thus, the Appellants' view of the materials newly presented in the final rejection are not part of the record. Accordingly, it is necessary to refute those materials and any significance attributed to them here.

Among the most important parameters that define a paper pulp are (1) fiber length, (2) brightness, and (3) the pulping process used.[15] A key step used to achieve a desired brightness is bleaching. It has been reported that a refining procedure activating a fiber surface before removal of fines had the best effect on the bleachability (*i.e.*, refining ***before***

[15] *See*, *e.g.*, http://www.paperonweb.com/pulppro.htm.

bleaching) and showed a decrease in bleaching chemical consumption.[16] Thus, Appellants respectfully submit that the sequence of steps for preparing a slurry of add-on material recited in the presently claimed process, *i.e.*, bleaching, drying, followed by milling of dried material, is neither taught nor suggested by the prior art.

Appellants further note that Gautam neither teaches nor suggests: (1) the use of dry grinding in papermaking, or (2) that wet and dry grinding are functionally equivalent processes. In contrast, as described in *A Critical Review of Current Theories for the Refining of Chemical Pulps*, Project 3384, Report Three: A Progress Report to Members of the Institute of Paper Chemistry, January 9, 1981, Pages 7-8,

> Refining of chemical pulp fibers differs from crushing ore in two respects: (1) the purpose of refining is not solely reduction of size and (2) refining is carried out under the plasticizing action of water. . . . It has been shown that dry grinding easily generates free radicals in the various chemical constituents of the cell wall. Thus, it is clearly established that water acts as a plasticizer and protective medium in the refining process.
>
> Because of the presence of water in refining and because of the structural features of the cell wall, one could very well state that the <u>main effect</u> <u>of</u> <u>refining</u> is an opening up of the fiber structure. ***That is not the case if the refining is done in air with a conventional low consistency refiner equipped with a knife tackle. Using normal bar clearance in such a refiner, the chemical pulp fibers are quickly physically and chemically decomposed without any development of internal or external fibrillation.***

(Underlining in Original; Bold Italics Added; Citations Omitted).[17] Fibrillating may be defined as a fine bleeding of fiber ends, resulting in a close-knit connection between individual fibers.[18] Similarly, as explained in U.S. Patent No. 5,385,640 (see Evidence Appendix),

> Mechanically beaten celluloses have long been employed in the paper and packaging industry. Chemi-thermomechanically refined wood pulps are typically dispersed in hydrobeaters and then subjected to wet refining in high speed disc mills. This level of structural manipulation as presently practiced is exclusively at the quaternary level. The objective of such processing is to disperse aggregated fiber bundles and increase available surface area for

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[16] *See* http://www.woodwisdom.fi/content/Old_Pdf/09_ww.pdf?from=4174734533604717.

[17] *See* http://smartech.gatech.edu/bitstream/1853/670/1/3384_003_071981.pdf.

[18] *See The Paper Making Process: From wood to coated paper*, the fifth technical brochure from Sappi Idea Exchange, at http://www.sappi.com/NR/rdonlyres/9053030F-70C1-439F-B159-634FF890D3F3/0/ThePaperMakingProcessEnglish.pdf.

> contact during drying to increase dry strength. Substantial size reduction and concomitant impairment of dewatering are undesirable and circumscribe the extent of processing. The measurement of the ease of water drainage from a beaten pulp is termed Canadian Standard Freeness and reflects the ease or rate of interstitial water removal from the paper stock.
>
> ***Finely ground or fragmented celluloses are well known. These products are produced by mechanical comminution or grinding of dried, refined cellulose. They are employed largely as inert, non-mineral fillers in processed foods and plastics.*** The manipulation is exclusively at the quaternary level of structure. It is achieved by application of a variety of size reduction technologies, such as ball and bar mills, high speed cutters, disc mills or other techniques described in part in U.S. Pat. No. 5,026,569. The practical limit of dry grinding is restricted in part by the thermal consequences of such processing on cellulose and in part to the economics of equipment wear and material contamination of the product. Micromilled cellulose (MMC) prepared in aqueous or other liquid media as described in U.S. Pat. No. 4,761,203 avoids the thermal decomposition associated with prolonged or intense dry grinding. This technique allows particle size reduction into the colloidal range (about 10 microns). It is believed to operate by indiscriminate micro-fragmentation of quaternary structure, without incurring the fusion/thermal degrading effects characteristic of dry grinding.

(Bold Italics Added; Column 2, Line 49 – Column 3, Line 17). Accordingly, while the prior art specifically discloses the use of wet grinding in papermaking, the prior art also specifically explains why dry grinding is not a suitable substitute for wet grinding in papermaking. Accordingly, the prior art teaches away from substituting dry grinding for wet grinding in papermaking. Thus, despite the arguments made in the final Office Action, one of ordinary skill in the art would not equate wet grinding and dry grinding in the papermaking art.

The final rejection also makes reference to "dry market pulp". But dry market pulp is a commodity used as feed stock to a paper mill – it does not represent a step used in further reducing the size of cellulose fibers laid down in a papermaking machine, or a further step in preparing add-on cellulose material. As noted above, both fiber length and brightness are two of the most important characteristics of paper pulp. Dry grinding which would shorten fibers would not be obvious to those skilled in the papermaking art – especially in view of its potential to adversely affect brightness due to discoloration associated with high friction and potential burning of fibers.

Appellants point out that the presently claimed processes are not directed to the production of dry market pulp. Rather, as noted above, the presently claimed method of

manufacturing a web having an applied pattern of add-on material comprises moving a base web along a first path; preparing a slurry of add-on material and repetitively discharging the slurry of add-on material upon the moving base web. The step of preparing a slurry of add-on material includes: cooking a fibrous cellulosic material, bleaching the material, pressing the cooked and bleached material to remove liquid, drying the pressed material, milling the dried material to produce fibers of a desired size, and mixing the milled material with water to hydrate the material and produce a slurry.

7. No *Prima Facie* Showing of Obviousness Has Been Made

Although Appellants do not concede that a *prima facie* case of obviousness has been made out in the rejections, rebuttal of a *prima facie* case of obviousness is merely "a showing of facts supporting the opposite conclusion." *In re Heldt*, 433 F.2d 808, 167 USPQ 676 (CCPA 1970). Facts established by rebuttal evidence must be evaluated along with the facts on which the conclusion of obviousness was reached, not against the conclusion itself. *In re Eli Lilly & Co.*, 902 F.2d 943, 14 USPQ2d 1741 (Fed. Cir. 1990); MPEP § 2142. "If rebuttal evidence of adequate weight is produced, the holding of prima facie obviousness, being but a legal inference from previously uncontradicted evidence, is dissipated. Regardless of whether the prima facie case would have been characterized as strong or weak, the examiner must consider all of the evidence anew." *In re Piasecki*, 745 F.2d 1468, 223 USPQ 785 (Fed. Cir. 1984).

Claim 1 recites a method of manufacturing a web having an applied pattern of add-on material including, *inter alia*: preparing a slurry of add-on material by cooking a fibrous cellulosic material, bleaching the material, pressing the cooked and bleached material to remove liquid, drying the pressed material, milling the dried material to produce fibers of a desired size, and mixing the milled material with water to hydrate the material and produce a slurry.

As explained above, "dry grinding" results in add-on material having a very narrow range of cellulose fiber sizes, and as a result the areas of the cigarette paper having the add-on material yield consistent and predictable performance. With this process, the add-on material is produced in much shorter time and consumes less energy than would be required to produce similar add-on material having a comparably narrow range of fiber sizes using multiple wet-slurry refining steps.

While Appellants respectfully submit that a *prima facie* case of obviousness has not been established, Appellants further respectfully submit that the improved results and more economical process associated with "dry grinding" in the claimed method, as compared to "wet grinding", also rebuts any possible *prima facie* case of obviousness.

Appellants again respectfully note that the newly claimed combination of steps provides important advantages; namely, (i) as outlined in the present specification, better control of fiber length for the add-on material, giving a more consistent and predictable performance of the banded paper (Page 3, Paragraph [0010], Lines 4-7), and (ii) savings in time and energy consumption during manufacturing operations (Page 3, Paragraph [0010], Lines 7-9, and at Page 6, Paragraph [0017]). Accordingly, the claimed combination both enhances the ultimate product and presents a novel combination of steps of making the ultimate product. Appellants respectfully submit that the claimed combination of steps and the advantages thereof are neither taught nor suggested by the prior art of record.

8. <u>Teaching Away</u>

A prior art reference must be considered in its entirety, *i.e.*, as a <u>whole</u>, including portions that would lead away from the claimed invention. *W.L. Gore & Associates, Inc. v. Garlock, Inc.*, 721 F.2d 1540, 220 USPQ 303 (Fed. Cir. 1983), *cert. denied*, 469 U.S. 851 (1984); MPEP § 2141.02.

Münchow discloses that: "In the preparation of paper, the raw material, i.e. wood pulp, wood, fine straw pulp or rag pulp, is admixed with paper pulp, fillers and pigments in order to achieve a closed surface and thus to improve the properties of the paper, especially the ***whiteness***, opacity and printability." (Column 1, Lines 23-27). And as Gautam explains, the preparation of slurry for production of cigarette paper initiates with cooking of flax straw feed stock, followed by a ***bleaching*** step and one or more refining steps, which are configured to achieve a weighted average fiber length in the flax slurry of approximately 0.8 to 1.2 mm. (Column 12, Lines 42-53). In conventional paper preparation, whiteness is a desired property.

In contrast, the present specification claims that add-on material to be applied in a pattern to a base web of cigarette paper is produced by cooking a fibrous cellulosic material, bleaching the material, pressing the cooked and bleached material to remove liquid, ***drying*** the pressed material, ***milling*** the dried material to produce fibers of a desired size and mixing

the resulting material with water to obtain the slurry of add-on material. (Page 4, Lines 1-6). Preferably, the milling step is configured to achieve a weighted average fiber length of approximately 0.5 to 1 mm. (Page 4, Lines 6-8). The bands of add-on material on the base web have fibers that are shorter than the fibers in the base web as a result of the processes performed on the add-on material. (Page 5, Lines 11-13).

The conditions involved with drying the pressed material, as well as milling or grinding the dried material, can cause discoloration of the material. In particular, heat introduced during drying, milling, and/or grinding can cause scorching of the material. Accordingly, in conventional preparation of paper[19], dry grinding is not used, because long fibers, which have not been scorched or degraded, are desired to produce white paper. Thus, the cited references teach away from the use of dry grinding, which can affect the desired whiteness in conventional preparation of paper.

For all these reasons, Appellants respectfully submit that the prior art teaches away from substituting dry milling for wet grinding in papermaking. Because there is no factual basis for asserting that dry milling is the equivalent of wet grinding, the process recited in Claims 1-5 would not be obvious to one of ordinary skill in the art on this record.

Thus, Appellants respectfully submit that the rejection of Claim 1 should be reversed and Claim 1 should be allowed. Claims 2 and 3 depend from Claim 1 and should also be allowed therewith.

B. Claim 4

Claim 4 pertains to a method according to claim 3 that further includes subjecting the flax straw feed stock to a process for removing non-fibrous components including shive before the step of cooking the fibrous cellulosic material.

The final Office Action asserts, "With regard to claims 4 and 5, the steps of removing shives and contaminants from a pulp is very well known and necessary step(s) after the cooking of the pulp", citing Chapter 9, Pages 98-132, of the *Handbook for pulp and paper technologists*, 2nd edition.

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[19] See, *e.g.*, (1) http://www.paperonweb.com/pulppro.htm;
(2) http://www.woodwisdom.fi/content/Old_Pdf/09_ww.pdf?from=4174734533604717;
(3) *A Critical Review of Current Theories for the Refining of Chemical Pulps*, Project 3384, Report Three: A Progress Report to Members of the Institute of Paper Chemistry, January 9, 1981; and
(4) *The Paper Making Process: From wood to coated paper*, the fifth technical brochure from Sappi Idea Exchange.

Claim 4 depends ultimately from Claim 1 and should be allowable therewith. But in addition, Appellants submit that the further basis for rejecting Claim 4 is untenable. Appellants respectfully submit that Chapter 9, Pages 98-132, of the *Handbook for pulp and paper technologists*, 2nd edition, deals with processing of pulps following the step of cooking. Figure 9-1, which is a schematic flowsheet for a kraft dissolving pulp mill, shows the following stations: a "Woodyard and Chipping" station followed by a "Cooking" station, then "Washing", "Screening I", "Bleaching", "Screening II", "Drying Machine", and finally "Finishing Department" stations. Furthermore, section 9.1, entitled "Defibering", begins, "All high-yield chemical and semichemical pulps must be defiberized by mechanical means following the cooking step." Similarly, the first paragraph of section 9.2, "Deknotting", ends with the sentence, "Knots are removed from the pulp prior to washing, and are either discarded as waste or returned to the digester infeed." Section 9.3 begins discussion of washing, specifically "Brown Stock Washing".

Appellants respectfully submit that Gautam does not disclose subjecting flax straw feed stock to a process for removing non-fibrous component including shive before the step of cooking the fibrous cellulosic material, as recited in Claim 4.

Accordingly, reversal of the rejection with respect to Claim 4 is respectfully requested.

C. <u>Claim 5</u>

Claim 5 also ultimately depends from Claim 1 and is allowable therewith. Claim 5 recites the method according to Claim 4, wherein the process for removing the non-fibrous component is preformed in a hammer mill.

Appellants respectfully submit that neither Gautam nor the *Handbook for pulp and paper technologists*, 2nd edition, discloses performing the process for removing the non-fibrous component in a hammer mill, as recited in Claim 5.

Accordingly, reversal of the rejection with respect to Claim 5 is respectfully requested.

VIII. Claims Appendix

Claims 1-5 as pending at the time of the final rejection are reproduced in the attached Claims Appendix.

IX. Evidence Appendix

The following materials are referred to elsewhere in this brief. Accordingly, copies of these materials are submitted herewith.

(1) http://www.paperonweb.com/pulppro.htm.

(2) *Targeted bleaching*, http://www.woodwisdom.fi/content/Old_Pdf/09_ww.pdf?from=4174734533604717.

(3) *A Critical Review of Current Theories for the Refining of Chemical Pulps*, Project 3384, Report Three: A Progress Report to Members of the Institute of Paper Chemistry, January 9, 1981, http://smartech.gatech.edu/bitstream/1853/670/1/3384_003_071981.pdf.

(4) *The Paper Making Process: From wood to coated paper*, the fifth technical brochure from Sappi Idea Exchange, http://www.sappi.com/NR/rdonlyres/9053030F-70C1-439F-B159-634FF890D3F3/0/ThePaperMakingProcessEnglish.pdf.

(5) U.S. Patent No. 5,385,640.

X. Related Proceedings Appendix

No known related proceedings exist for this appeal. Accordingly, no Related Proceedings Appendix is included herewith.

Respectfully submitted,

BUCHANAN INGERSOLL & ROONEY PC

Date August 16, 2007

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VIII. CLAIMS APPENDIX

The Appealed Claims

1. A method of manufacturing a web having an applied pattern of add-on material, said method comprising:

moving a base web along a first path;

preparing a slurry of add-on material; and

repetitively discharging said slurry of add-on material upon said moving base web, said step of preparing a slurry of add-on material including:

cooking a fibrous cellulosic material,

bleaching the material,

pressing the cooked and bleached material to remove liquid,

drying the pressed material,

milling the dried material to produce fibers of a desired size, and

mixing the milled material with water to hydrate the material and produce a slurry.

2. The method according to claim 1, wherein said step of repetitively discharging said add-on slurry comprises:

continuously moving a belt having an orifice along an endless path, said belt moving step including the step of moving said belt along a first portion of said endless path where said orifice is communicated with a reservoir so as to discharge said add-on slurry from said reservoir through said orifice onto said base web as said orifice traverses said first path portion.

3. The method according to claim 1, wherein said fibrous cellulosic material comprises flax straw feed stock.

4. The method according to claim 3 further including:

subjecting the flax straw feed stock to a process for removing non-fibrous component including shive before the step of cooking the fibrous cellulosic material.

5. The method according to claim 4, wherein the process for removing the non-fibrous component is preformed in a hammer mill.

IX. EVIDENCE APPENDIX

PROPERTIES OF PULP

The three most important Parameters which defines the pulp are. 1.) Fiber Length, 2.) Brightness, 3.) Pulping process used. e.g. Northern Soft Wood Bleached Kraft (NSWBK). The Northern Soft Wood tells it is long fiber pulp. Bleached tells, it has high brightness and Kraft tells that Kraft (Sulfate) pulping process is used to produced this pulp. Similarly Southern Hard Woof Unbleached Kraft, will be a short fiber wood unbleached (low brightness) pulp made by kraft process. Link to a few typical Market Pulp data sheets

http://www.ariver.com/HardwoodDataSheet.pdf

http://www.ariver.com/HardwoodDataSheet.pdf

Ash Content in Pulp

Ash content in pulp may consists of various chemicals used during pulping/bleaching, mineral matter from wood or metallic matter from pipes and other machinery. It is not important parameter of pulp.

Ash is the residue left after igniting pulp at 525 ^{0}C (As per TAPPI T211). Ash is reported in % of residue to dry pulp basis.

The standard procedure of measuring ash content is laid out in TAPPI T211, ISO 1762

Brightness of Pulp

Brightness of paper is discussed in Paper Properties. The paper brightness is mainly dictated by pulp brightness. There are some modification in stock preparation which can alter paper brightness to some extent such as filler, sizing, whitening agent, dying etc. In short

Conductivity of Pulp

Electrical grade papers such as cable paper, condenser tissue or insulation paper etc., require very low conductivity to electricity. The presence of metal ion more specifically iron ion contribute to pulp conductivity The pulp used for electrical grades are washed with demineralized water, beater or refiner use lava or other non-metallic bars and contacting surfaces of all equipment are made of stainless steel.

Values for the conductivity of the water extract of the pulp are expressed in μS/m.

S = **Siemen** (SI unit of **electric conductance**) = 1 **mho**.

Dirt in Pulp

Dirt content of pulp particularly of recycled pulp is important for its suitability to make fine paper. Dirt is any foreign material in pulp. TAPPI defines dirt as foreign matter in a sheet which, when examined by reflected, not transmitted light, has a marked contrasting color and has an equivalent black area of 0.04 mm^2 or more.

The standard procedure of measuring dirt content is laid out in TAPPI T213

Drainage Time of Pulp

Here the drainage time of pulp is discussed in reference to market pulp and/or unrefined pulp. The drainage time of pulp or freeness or slowness of pulp is modified to have some desired properties in the paper, here that is not discussed.

Drainage of unrefined pulp which is measured as freeness can give an indication on : 1) Fiber Length of pulp, as long fiber pulps have more freeness compared to short fiber pulps, 2) Damage to fiber during pulping, bleaching or drying as short fibers or fines produced during pulping operation reduces pulp freeness, 3) Refining energy required to achieve certain slowness during stock preparation.

The standard procedure of measuring pulp drainage is laid out in TAPPI T221, T227, ISO 5267-1 and ISO 5267-2

Dry Content of Pulp

Consistency: is the term used to describe solid content of pulp during pulp processing. For pulp and paper maker this is **the most important** process parameters. All equipments are designed to handle pulp at and up to certain consistency. Pulp consistency is roughly

divided in to three ranges:

Low Consistency: <5%
Medium Consistency: 5 - 15%
High Consistency: >15%

It is the desire of every pulp maker to keep pulp at the highest possible consistency to minimize dilution water usage and which ends up as effluent. Higher consistency also helps in reducing the bleaching chemical consumption. But there are practical limitation of handling pulp at higher consistency such as high viscosity which make pulp flow very difficult.

The standard procedure of measuring pulp consistency (up to 25%) is laid out in TAPPI T240.

Moisture Content of Market Pulp: is important from storage, transportation and handling point of view. Most of the market pulp are sold, stored, transported and used as air dry. The useable part of pulp is dry fiber only, so the tendency is to minimize the moisture content op pulp.

Small quantity of pulp is sold as wet lap also. Wet lap pulp is not dried at source and transported at about 50% moisture content. It is feasible for short distance transportation and if pulp is to be used immediately at user end.

Extractives (Low Molecular Weight Carbohydrates) in Pulp

The low molecular carbohydrates indicates an extent of cellulose degradation during pulping and bleaching process, which may effect pulp strength and other properties. Pulp is treated with 1% hot NaOH solution for one hour to estimate loss of yield due to extractives.

The standard procedure of measuring 1% Hot Alkali Solubility is laid out in TAPPI T212

Fiber Length of Pulp

Length of fibers (arithmetic average, weighted average etc.) is one of the most important parameters of pulp. Pulp strength is directly proportional to fiber length and dictates its final use. A long fiber pulp is good to blend with short fiber pulp to optimize on fiber cost, strength and formation of paper. Softwood with pulps in general have longer fiber compared to hard wood pulp. Pulp made from woods grown in cold climate in general have longer fiber compared to wood grown in warmer climates.

Chemical pulps in general have higher fiber length compared to semi chemical pulp and mechanical pulp, when made from same wood. More fibers get damaged/shorten by mechanical action than chemical action.

There are several method to measure /report fiber length of pulp. The 'fiber length of pulp by projection' is described in TAPPI T232. The 'fiber length of pulp by classification' is described in TAPPI T233. "Fiber length of pulp and paper by automated optical analyzer using polarized light' is described in TAPPI T271.

The coarseness of pulp fiber is described in TAPPI T234.

Kappa Number of Pulp

Kappa number is determination of relative hardness, bleachability or degree of delignification of pulp. It is important parameter of unbleached pulp which is to be bleached.

The method to find kappa # of pulp is described in TAPPI T236.

Pulping Process

Though the pulping process used is directly not a pulp property but this is one of the most important parameters used in specifying the pulp. As we move from full mechanical to full chemical pulping process, strength of pulp and bleachability improves. Strength improves due to less degradation of fibers and bleachability as more lignin is removed in chemical than mechanical pulping processes.

Yield: Pulp yield is mainly govern by the pulping process. Mechanical pulping processes which provide high yield, retain almost all constituents of wood. Lignin which is second highest to cellulose, does not bond to itself or cellulose fibers as fibers do, don't contribute to any bonding, resulting in weak pulp. Secondly lignin is brown in color and to maintain high yield of bleached pulp, lignin is not removed during bleaching, but only chemically modified.

Tensile Strength of Pulp

This is not the tensile strength of individual fiber, which is even higher than or comparable with steel. The tensile strength discussed here is maximum strength of randomly oriented pulp fiber when formed in a sheet. This tensile strength gives an indication of the maximum possible strength of pulp beaten under ideal condition. This again an indication of what level of tensile strength can be achieved in real paper making environment.

One way of measuring tensile strength of pulp is "zero span breaking strength' described in TAPPI T231. Wet zero span tensile strength of pulp is measured using TAPPI T273.

Viscosity of Pulp

Solution viscosity of a pulp gives an estimation of the average degree of polymerization of the cellulose fiber. So the viscosity indicate the relative degradation of cellulose fiber during pulping /bleaching process.

Dissolving pulps from wood, which contains a large proportion of alpha cellulose, give higher viscosity values than paper pulps.

The standard procedure of measuring pulp viscosity is laid out in TAPPI T230

TOP OF THE PAGE

Targeted bleaching
Täsmävalkaisu

Coordinator Bo Hortling
Koordinaattori

Total project budget / Wood Wisdom funding
Kokonaiskustannus / Wood Wisdom -rahoitus
€ 926 715 / 516 338

1) The effect of the inhomogeneous chemical structure of the fibre wall on the delignification (Targeted bleaching)
Kuituseinän epähomogeenisen kemiallisen rakenteen vaikutus delignifioitumiseen (Täsmävalkaisu)

KCL
Bo Hortling
8/1997–8/2000
Tekes € 228 736

2) Enzymes in the activation of lignin
Entsyymit massan ligniinin aktivoinnissa

VTT
Raija Lantto
8/1997–8/2000
Tekes € 116 050

3) Solid-state NMR studies of cell wall components
Kuituseinän komponenttien tutkiminen kiinteän olomuodon NMR:llä

University of Helsinki
Helsingin yliopisto
Sirkka Liisa Maunu
10/1997–12/2000, I
1/2001–6/2002, II
Tekes € 171 552

Abstract

The results showed that it is possible to activate the bleaching processes by specific enzymes attacking galactans in the pulps and by refining treatments. The effects obtained were, however, smaller than expected. The most pronounced effect for the enzymatic treatments was obtained for unbleached pulp. The removal of fines after refining decreased the lignin content and decreased the consumption of bleaching chemicals. New knowledge about the properties and structures of fibres and fibre wall components was obtained. Especially the work with solid-state NMR gave new information about the insoluble fibre fractions and components.

Tiivistelmä

Tulokset osoittivat, että valkaisuprosessia on mahdollista aktivoida spesifisillä entsyymeillä, jotka vaikuttavat massojen galaktaaneihin, sekä jauhatuskäsittelyillä. Saadut vaikutukset olivat kuitenkin odotettua vähäisempiä. Parhaat vaikutukset saatiin valkaisematonta massaa käytettäessä. Hienoaineen poisto jauhatuksen jälkeen vähensi ligniinipitoisuutta ja valkaisukemikaalien kulutusta. Uutta tietoa saatiin kuitujen ominaisuuksista ja rakenteista sekä kuituseinäkomponenteista. Erityisesti kiinteän olomuodon NMR antoi uutta tietoa liukenemattomista kuitufraktioista ja komponenteista.

1 Introduction

1.1 Background

The composition of the fibre surface differs from the rest of the fibre, which is believed to render delignification and bleaching difficult. By isolating fibre fractions (long fibres and corresponding fines) information is obtained about their effect on the physical properties of the fractions and by characterising their components also about their chemical and physical properties.

In earlier projects, methods had been developed for characterisation of components in the fibre wall, and it was also suggested that there occurs a residual lignin-galactan complex of high molar mass, which makes the delignification difficult.

1.2 Objectives

The objective of the study was to find enzymatic and mechanical treatments, which split the lignin-galactan complexes in order to improve the bleaching procedures. In addition analytical methods were developed to obtain new information about the fibre fractions and the components in the fibre fractions (fines and corresponding long fibres). Especially the solid-state NMR will be applied in order to obtain new information about the structures and interactions of the fibre components.

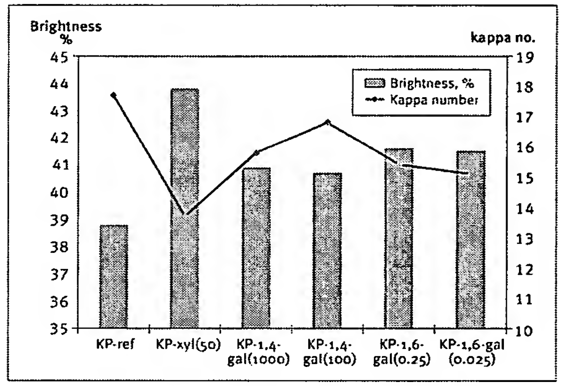

Figure 1. The effect on the oxygen delignification of the treatments with galactanases of kraft pulp is seen as changes in kappa number and brightness. The enzymatic treatment was performed at a consistency of 5% at pH 5 at 40°C for 20 h. The enzyme charges were 100 or 1000 nkat/g for 1,4-galactanase, 0.25 or 0.025 mg/g for 1,6-galactanase and 50 nkat/g for xylanase.

2 Results and discussion

The enzymatic and mechanical activation of spruce kraft pulps towards bleaching

Lignin-galactan complexes occurring in the outer layers of the fibre wall were assumed to hinder selective delignification of unbleached pulps. The activation of these complexes was performed by splitting the galactan chains using specific galactanases or by refining procedures activating the surface of the fibres.

Activation by enzymes

Spruce kraft pulp (KP) was treated before the oxygen delignification with different charges of 1,4- and 1,6-galactanases. Compared to the reference pulp, the increase in brightness was about 2 units higher when 1,6-galactanase was used and 1.5 units higher when 1,4-galactanase was used (Figure 1). A decrease in the enzyme charge did not change the activation effect significantly and the yield losses after the treatments were very low. Reference treatments were performed with xylanases, which gave a higher increase in brightness and decrease in kappa number, but more significant yield loss. The positive effects obtained with the galactanases were not large enough for practical use at the moment.

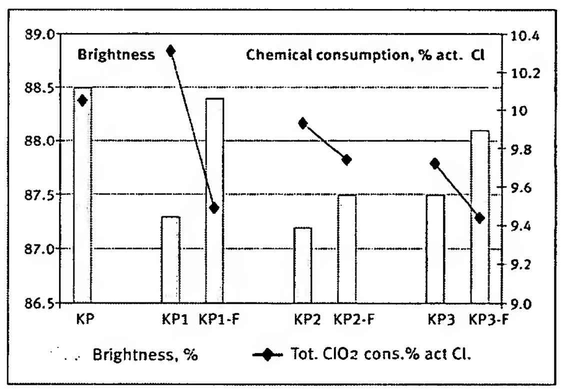

Figure 2. The brightness values and ClO_2 consumption (% act.Cl) after the DEDED sequence of the pulps after refining and after removal of fines (F). KP = starting pulp, KP1 = low edge load in water, KP2 = high edge load in water and KP3 low edge load in 0.01 M NaOH.

Activation by refining

The refining was performed at a lower (surface activating) and a higher (fibre cutting) edge load. The effect of refining on the pulps was monitored by taking samples at certain intervals. The first sample was taken before the refining started after a 10 min pulpering period and then three samples were taken during the refining for isolation and characterisation of fines and peeled fibres. The content of lignin in fines was twice that of the pulp and the content of extractives was more than two times higher than for the pulp. Smaller differences were seen between the composition of carbohydrates.

The refining did not improve the bleachability during an ECF sequence and the chemical consumption was even higher than for the unrefined pulp. However, the removal of fines decreased the chemical consumption, apparently due to the lower lignin content of the peeled fibres in the pulps. The changes in brightness and chlorine dioxide consumption after the enzymatic treatment of the spruce kraft pulp followed by a DEDED sequence is given in Figure 2.

The results indicated a clear decrease in chlorine dioxide consumption after refining and removal of the fines from the differently refined pulps. The best results were obtained by activation with a surface active refining in water after removal of the fines. In this case the consumption of chlorine dioxide decreased from 10.3 %actCl to 9.5 %actCl.

Results of sequential refining

The sequential refining was performed in order to obtain additional information about the structures and properties of the fibre wall layers and it was different from the refining used in the activation procedure, where the fines were present during the whole refining procedure. In sequential refining the fines were removed before each subsequent refining step, Figure 3.

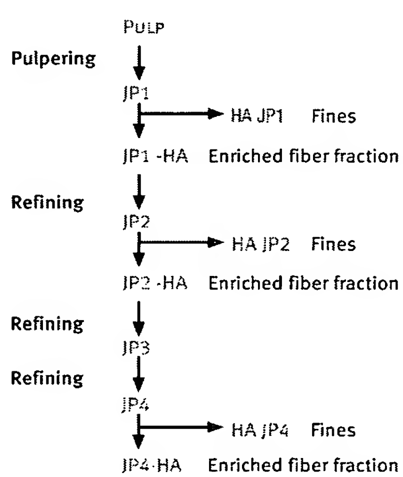

Figure 3. Sequential refining of the pulp using an edge load of 2.5 Ws/m and an energy of 70 kWh/t at each refining point. JP1-4 represent the refining points, HAJP1-4 the fines removed and JP1-4-HA the peeled fibres.

Properties and structures of the fibre fractions and their components

Properties of the fibre fractions

During the surface active refining no changes in the fibre dimensions were observed. The yields of the fibres obtained by sequential refining were higher than the yield obtained by the conventional refining. The fines were investigated more in detail after the sequential refining and it was seen that a significant decrease in the content of ray cells or plate-like particles occurred when the refining was proceeding. The increase in the swelling speed of the fibre wall measured in EWNN also increased as the sequential refining proceeded. According to the ordinary ^{13}C CPMAS and delayed contact measurements, the cellulose crystallinity is lower in fines than in long fibres. However, after chemical removal of lignin and hemicelluloses, the cellulose crystallinity as well as the lateral fibril dimensions were found quite similar in fines and fibre fractions. In the refined fibre fraction the lateral fibril aggregate dimension was found slightly higher than in the original kraft pulp. This was also thought to be an indication of better swelling properties of the refined fibres.

Structures and properties of fibre wall components

The structures of the fibre wall components, especially the residual lignin-carbohydrate complexes, were characterised by chemical, spectroscopic and pyrolysis methods. By characterising native lignins it was seen that apparently native lignin- galactan linkages were left in the residual lignin-carbohydrate complexes. The lignin structures were not changed during the activation treatments, but an enrichment of 1,3-galactan structures was observed in the peeled fibre fraction compared to the enrichment of 1,4-linkages in the primary and secondary fines fractions. Also the content of acidic groups was higher in the fibre fractions. According to the CPMAS measurements the insoluble residual lignin fractions obtained after protein purification contain considerable amounts of carbohydrates. Some of these carbohydrates are apparently unhydrolyzed cellulose, which could be an indication of some strong interactions between lignin and cellulose. In the insoluble residual lignin fractions isolated from the fines the amount of unhydrolyzed cellulose is clearly higher than in the residual lignin of fibres or original pulp. The assumed inter-

actions between lignin and cellulose may thus be stronger in fines and especially in primary fines.

The residual lignin in the fines had a higher molecular size than the lignin in the peeled fibre fraction and it was also slightly more condensed, but in other respects the lignin structures were similar. After oxygen delignification and bleaching procedures the residual lignin obtained a more hydrophilic structure and also a decrease in molecular size occurred, which is in accordance with earlier results. NMR results showed that after oxygen delignification the residual lignin structure is more condensed than in the unbleached pulp.

3 Conclusions

The mechanical and enzymatic activation treatments of the pulps were developed in order to improve the bleachability of spruce kraft pulps. The changes of the fibres and fibre wall components were monitored in order to develop the activation treatments and to obtain new knowledge. Various solid-state NMR techniques were also applied successfully in the studies of the lignin and pulp samples.

A refining procedure activating the fibre surface before the removal of fines had the best effect on the bleachability and was seen as a decrease in chemical consumption. The decrease in chemical consumption is due to the decrease in lignin content when the lignin-rich surface material was removed. The specific 1,4- and 1,6-galactanases gave a slight improvement in the bleachability of the pulp without significant yield losses. However, it was shown that also 1,3-galactan linkages occurred in the fibre wall. The 1,3-galactanase was not available and its effect on the bleachability was thus not tested. Proteases had a boosting influence on the bleaching effect of xylanases. The results obtained confirm the hypothesis that the use of surface-activating treatments, like specific galactanases and proteases, lead to improvements in pulp bleachability, with only marginal yield losses and thus a decrease in bleaching chemical costs. Further development of specific enzymes is still, however, needed. Surface activation by refining is another way of activation of kraft pulps towards bleaching. Removing the fines reduced the consumption of bleaching chemicals.

According to the structure and properties of the isolated residual lignin it should have been dissolved during pulping. This was not the case, which supports the idea that linkages between lignin and polysaccharides, which were also detected by solid-state NMR, are one important reason for the incomplete delignification. A part of the linkages occurred already in the wood. The galactanase activation of the pulp was seen as a slight decrease in the galactose content of the isolated residual lignin-carbohydrate complexes.

The residual lignin of the fines fractions was sparingly soluble in alkali, partly due to high molar mass compared to the residual lignin in the peeled fibres and/or stable linkages to polysaccharides. However, no significant changes in the lignin structure were seen during the enzymatic and mechanical treatments by chemical and pyrolysis measurements. No significant differences were seen on the basis of the NMR measurements, either.

In order to obtain additional information about the structures and properties of the fibre wall and its components the sequential refining method was developed in order to characterise fines and corresponding peeled fibres from different layers of the fibre wall. The lignin and extractive contents were highest in the primary fines and decreased towards the end of the refining. The carbohydrate compositions were similar in all fibre and fines fractions except for the slight enrichment of the hemicelluloses in the primary fines. Uronic acids were enriched in the long fibre fraction.

It was seen that the removal of fines material at the end of the sequential refining increased air penetration, which is beneficial e.g. for sack paper grades.

4 a) Capabilities generated

The project generated new methods for isolation and characterisation of fibre fractions (fines and corresponding peeled fibres) and its components. New knowledge about the structures and properties of the fibre fractions was obtained, which can be used in understanding and developing delignification processes.

Degrees
One doctoral thesis is under preparation in the subproject dealing with solid-state NMR spectroscopy.

b) Utilisation of results

The results have been distributed to the companies taking part in the KCL-STFI joint chemical pulp research programme and to the participants of the Wood Wisdom programme. They will also be used in new projects concerning delignification reactions and pulp properties. Subproject 3 on NMR is continued as the Fiberface by NMR project (1.1. 2001-30.6.2002).

5 Publications

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*Liitiä, T., Maunu, S.L. and Hortling, B., Solid State NMR Studies on Inhomogeneous Structure of Fibre Wall in Kraft Pulp (accepted to be published in Holzforschung)

Hult, E.-L., Liitiä, T., Maunu, S.L., Hortling, B. and Iversen, T., A CP/MAS ^{13}C-NMR study of cellulose structure on the surface of kraft pulp fibres (submitted to Carbohydrate Polymers)

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T. Liitiä, S. Maunu, B. Hortling, Solid state NMR studies of residual lignin, 10th International Symposium on Wood and Pulping Chemistry, Yokohama, Japan, June 7-10, 1999, Volume I, 358-363.

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*Tamminen, T., Hortling, B., Ranua, M., Luonteri, E., Suurnäkki, A., Tenkanen, M. and Buchert, J. Enhanced bleachability of spruce kraft pulp by mechanical and enzymatic treatments,10th International symposium on wood and pulping chemistry - 10th Biennial ISWPC - Main symposium, Yokohama, Japan, 7-10 June 1999, vol. 1, Oral presentations, pp 584-588.

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6 Cooperation

Advisory group

Osmo Keitaanniemi, Metso Chemical Pulping, chair
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The lateral fibril and fibril aggregate dimensions determined by solid-state NMR were conducted in collaboration with Eva-Lena Hult and Tommy Iversen in STFI.

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Tiina Liitiä

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A CRITICAL REVIEW OF CURRENT THEORIES FOR THE REFINING OF CHEMICAL PULPS

Project 3384

Report Three
A Progress Report
to
MEMBERS OF THE INSTITUTE OF PAPER CHEMISTRY

January 9, 1981

THE INSTITUTE OF PAPER CHEMISTRY

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THE INSTITUTE OF PAPER CHEMISTRY

Appleton, Wisconsin

A CRITICAL REVIEW OF CURRENT THEORIES FOR THE REFINING OF CHEMICAL PULPS

ABSTRACT

The literature dealing with effects of refining on the structure of individual cellulose fibers is reviewed with an emphasis on creating a universal list of primary effects of beating. The various theories and hypotheses put forward to explain the mechanics of refining are critically reviewed. Less relevant material of this latter subject seems to be avilable, and much of the fundamental and still valid research work in the area is quite old. New experimental evidence is presented in support of a hypothesis that initial stages of refining consist of treatment of flocs instead of individual pulp fibers. The significance of this postulate to refining research is discussed.

The review is partially based on the literature study done by the author during the summer of 1979, when he was a visiting scientist at The Institute of Paper Chemistry. It was first presented at the International Symposium on the Fundamentals of Refining, held at IPC September 16-18, 1980.

ACKNOWLEDGMENT

The author is indebted to The Institute of Paper Chemistry for arranging the visit. The help of the IPC staff working on the refiner research project is also gratefully acknowledged.

INTRODUCTION

The term "Refining of chemical pulp," in connection with papermaking, means mechanical treatment of the chemical pulp fibers in order to render them more suitable for the papermaking. During this review the terms refining and beating are used synonymously. This review is restricted to low consistency mechanical treatment of low and medium yield chemical pulps.

Due to the very high amount of technical literature dealing with various aspects of refining[1], it has been impossible to review all the "current" refining literature. Instead, a method of selective emphasis has been used.

Based on the present information concerning the structure of paper, it is possible to define refining as the process of creating desirable structural changes in the cell wall of the pulp fibers at the expenditure of mechanical energy (Fig. 1) (1). The nature and extent of the desirable structural changes depends very much on the end use properties of the paper grade in question and on the papermaking quality of the unrefined pulp fibers. Unfortunately, the present refining or beating processes also create simultaneously unwanted structural changes, i.e., damage in the pulp fibers. Thus, in the refining process a compromise has to be made between the wanted and the attainable effects of refining on the pulp fibers and on the sheet characteristics. As can be seen from Fig. 1, the papermaker has a large number of processing variables available to affect the result of the refining process. Unfortunately there seems to be no generally accepted theory of refining that would tell the papermaker what is the exact effect of the various controlling variables (shown in Fig. 1) on the structural changes obtained, i.e., on the output of the refining process. Besides, in typical industrial refining, for instance in

[1] About 30-50 major references per year are listed in the annual index of ABIPC.

integrated papermaking, the only active controlling variable of the refining process is the load adjustment.

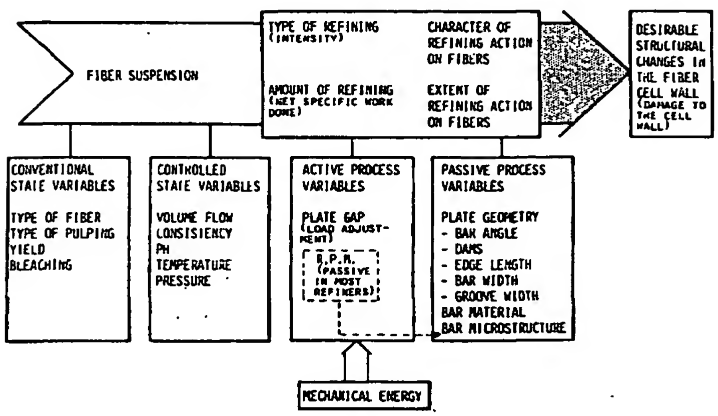


Figure 1. Qualitative Model of Refining Process

The lack of a generally accepted theory of refining may partially be due to the lack of suitable measurement techniques to characterize the structural changes in the papermaking fibers due to refining (2). However, the major reason seems to be the practical need for a shortcut in the chain of reasoning of how to control the quality of the paper through refining (Fig. 2).

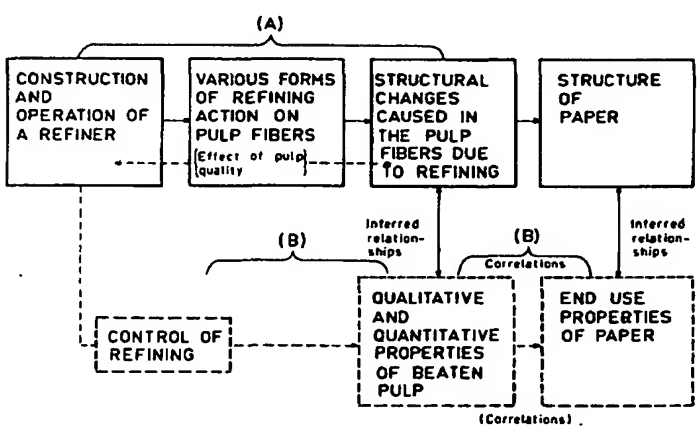


Figure 2. Schematic Representation of the Scope of Theory of Refining (A) and the Area of Common Refining Research (B)

Another reason for the lack of an acceptable theory of refining is probably the fact that since paper is a composite structure, all the structural needs dictated by the end use properties of the paper cannot usually be satisfied through refining of the fibers. Instead a compromise often has to be made between the various structural changes of the fibrous cell wall that are caused by refining. The requirement for an efficient use of mechanical energy in refining also affects the compromise to be selected.

As can be seen from Fig. 2, much of the refining research in the past has been along the lines of pulp evaluation, although in recent years a considerable amount of research has also been directed towards characterizing the refining process. As a result of such characterizing research, technical possibilities to control the refining process have been somewhat advanced.

The increased knowledge concerning the structure of paper[1] and how that is affected by the structure and chemical composition of the component fibers has made it possible to deduce - at least to a certain extent - what the desirable structural changes in the cell wall of the component fibers are in refining. In addition, there also is information about how the unwanted structural changes, i.e., damage to the cell wall, affects the structural behavior of paper in the various end use applications. There seems to be a general agreement of what are the primary structural changes caused by beating or refining (10-15), although the emphasis on what are the important refining actions on fibers seems to generate disagreement between the representatives of the various schools of thoughts (16).

In comparison to the cited advancement of information concerning (a) the structure of paper and how that is affected by the structure of the component

[1]Summed up for instance in the Proceedings of the Fundamental Research Symposiums organized by RPBMA (nowadays BPBIF) (6-9).

fibers, and (b) the primary changes in the cell wall structure caused by refining, very little relevant information has been published over the years about the mechanism (action) that causes the various primary effects of refining in the pulp fibers[1]. In other words, it is not known how the mechanical energy of refining is transferred to the fibers, i.e., what is the behavior of the stock in the refining zone and what is the path of fibers through the refiner. It is believed that as long as this piece of information is lacking, there will be no grounds for the establishment of a "good" theory of refining.

If such a theory were available, it could be used, for instance, in the following cases:

1. Design and selection of the best possible operating conditions for a new refiner that would (see Fig. 2)
   - have a higher energy efficiency than the present refiners
   - be applicable to refining of new types of pulp fibers
   - generate new combinations of papermaking properties into refined pulp fiber, i.e., combinations that are unattainable with present refiners.
2. Choice of the optimal process conditions for an existing refiner taking into account the process modifications that are easily carried out in the process of refining (Fig. 1).

The expenditure of electrical energy in refining of chemical pulp fibers is around 720-1800 MJ/t. pulp (200-500 kWh/t.). For a papermill producing about 100,000 t./a, the direct energy cost of refining will easily mount to about 0.3-0.6 million U.S. dollars annually. Although this sum is not a very large manufacturing cost on a relative basis, it represents a considerable potential for energy saving and for

[1]This same fact was emphasized also by Atack in his 1977 review of beating (15).

profit making. This is so because out of the total cost of refining, including capital cost and replacement cost of the worn-out tackle or plates, the role of direct energy consumption is dominant, i.e., about 80% (17,18).

Thus the savings obtained can easily pay back an investment for a new set of refining tackle or for a change in the rotational direction of the rotor.

PRIMARY EFFECTS OF REFINING ON FIBERS

For a thorough analysis of this subject matter one is referred to the book by Emerton (11) and to the reviews by Higgins and Yong (12), Fahey (14), Atack (15), and Clark (16). Only a concise representation of the effects of refining on the structure of the fibers is given here based on an earlier review of the matter (13) and supplemented with selective references to later publications.

It is somewhat difficult to define the primary beating effects on the structure of the fibers since fibers themselves form a very heterogeneous source of material. One needs only to point out that fibers in a given pulp have a large distribution of sizes (length, diameter, thickness of the cell wall, fibrillar angle) and that the chemical composition of the cell wall and distribution of the main chemical constituents through the cell wall vary considerably. Besides, the unrefined fibers already have some structural damage in their cell walls (92).

We can define the primary effects of refining as such changes in the structure of the fiber; with these effects it is - at least in theory - possible to differentiate between a refined fiber and an unrefined one. A further requirement for the primary effect of refining is that it cannot be divided into components. However, there is no need to restrict the appearance of the primary beating effects on the fibers to only one type of an effect per a particular fiber. On the

contrary, due to the fact that refining is carried out in water, and due to the mode of transferring mechanical energy into the fibers during refining, it is easy to visualize that many of the primary beating effects do in fact occur simultaneously in a given fiber.

Besides being of fundamental nature and of simultaneous occurrence, the primary beating effects have to be defined also as irreversible structural changes (19). Because the refining process, i.e., the mechanism of transferring mechanical energy into fibers and creating primary beating effects in the fiber, is controlled by some type of probability function (4) and because of the heterogeneous flow pattern through the refiner (20), the distribution and extent of the primary beating effects among the refined fibers is very heterogeneous. In other words, it is possible to find fibers that have received practically no refining treatment and fibers that have received refining action well over the average amount (21-23). Besides, the type and extent of refining treatment within the fiber is localized (92). One may also reach conclusions regarding the heterogeneous nature of the refining process (24) based on the change of the fiber length distribution curve during refining, i.e., that after considerable refining and cutting of fibers, it is still in many cases possible to find "uncut" original fibers in the stock.

ROLE OF WATER IN REFINING

Refining of chemical pulp fibers differs from crushing ore in two respects: (1) the purpose of refining is not solely reduction of size and (2) refining is carried out under the plasticizing action of water (25). Since the pioneering study by Kress and Bialkowski (26), there have been other studies made in order to find out the mechanism of water in refining (27-29). It has been shown that dry grinding easily generates free radicals in the various chemical constituents of the cell wall

(30). Thus it is clearly established that water acts as a plasticizer and protective medium in the refining process.

Because of the presence of water in refining and because of the structural features of the cell wall, one could very well state that the main effect of refining is an opening up of the fiber structure. That is not the case if the refining is done in air with a conventional low consistency refiner equipped with a knife tackle (31). Using normal bar clearance in such a refiner, the chemical pulp fibers are quickly physically and chemically decomposed without any development of internal or external fibrillation.

CLASSIFICATION OF THE PRIMARY BEATING EFFECTS

The old refining literature usually listed three primary beating effects, i.e., structural changes in the fibers due to refining. These are: (1) cutting and/or splitting of the fibers, (2) external fibrillation of the fiber surface and (3) hydration of the cell wall material. A recent review of the mechanical treatment of chemical pulps (14) speaks about only two refining actions: (a) breakage of intrafiber hydrogen bonds and replacing of them with fiber-water hydrogen bonds, and (b) breakage of covalent bonds.

Table I summarizes the primary effects listed by various authors (12-14, 16,32). As can be seen, disagreement between the depicted lists is small. It is a matter of taste if the production of fines needs to be listed as a separate primary beating effect[1]. Due to the heterogeneity of the chemical pulp and because of the basic structure of the cell wall, it is to be expected that all the listed effects occur simultaneously, but the extent of their occurrence can vary considerably. It is, however, difficult to decide which of the listed primary beating effects is

[1]The order of presentation of the effects by no means implies any preferable order of significance or order of probability of occurrence.

TABLE I. PRIMARY BEATING EFFECTS (STRUCTURAL CONSIDERATIONS)

HISTORICAL	FAHEY (14)	HIGGINS AND YOUNG (12)	GIERTZ (32)	CLARK (16)	EBELING (13)
CUTTING/ SPLITTING	BREAKING OF COVALENT BONDS	FIBER SHORTENING	CUTTING AND CRUSHING	SHORTENING	FIBER SHORTENING
EXTERNAL FIBRILLATION		EXTERNAL FIBRILLATION	SUCCESSIVE CLEAVAGE OF EXTERNAL LAYERS OF THE CELL WALL AND SUBSEQUENT BREAKING AWAY OF THESE LAYERS	EXTERNAL SPLITTING	SUCCESSIVE CLEAVAGE OF EXTERNAL LAYERS OF THE CELL WALL AND THEIR SUBSEQUENT BREAKING AWAY *
HYDRATION	INTRAFIBER H-BOND BREAKING	INTRAFIBER H-BOND BREAKING	INTRAFIBER H-BOND BREAKING	INTERNAL SPLITTING	DELAMINATION OF INTERNAL CELL WALL LAYERS *
		PRODUCTION OF FINES		(PRODUCTION OF DEBRIS) *	(* = SEE ABOVE)
			CREATION OF DISLOCATIONS	(LONGITUDINAL COMPRESSION) *	LOCAL DISLOCATIONS OF THE CELL WALL STRUCTURE
				* SECONDARY EFFECTS	DISSOLUTION OF THE CHEMICAL COMPONENTS OF THE CELL WALL AND SIMULTANEOUS FORMATION OF COLLOIDAL CARBOHYDRATE SOLUTION ON THE SURFACES AFFECTED

few results which show that part of the cell wall material gets dissolved during refining (26,38,49-53). This may be taken as indirect evidence for the appearance of molecular fibrillation, since molecular fibrillation is a prerequisite for the complete dissolution of the polymeric components of the cell wall. The importance of various cations on the pulp and paper properties (54) may also be taken as an indication of the presence of molecular fibrillation. Similarly, the increased beating response of high yield pulps and of recycled old corrugated containers (55,56) due to ozone treatment would seem to indicate the presence of molecular fibrillation at least on the external fiber surfaces. Besides, many researchers, in the area of refining, have emphasized "molecular fibrillation" as one of the most important refining results (11,26,32,38,40,60-63). When speaking about molecular fibrillation and its role in refining and papermaking, one should also keep in mind that it is very difficult if not impossible to beat a high alpha-cellulose pulp.

Creation of New Particles

This class of primary beating effects can also be divided into three subclasses based on the size of structural units involved: (a) cutting[1] of fibers, (b) cutting and/or splitting loose of cell wall lamellae and macrofibrils, and (c) dissolution or cutting away of the polymeric molecules of the cell wall. The cutting of fibers and detachment of large parts of lamellae is called "generation of fines" and there is plenty of experimental evidence about this primary effect of beating. The detachment of lamellar and macrofibrillar matter from the cell wall is often called "generation of crill," and there is also plenty of experimental evidence about it since the introduction of the term by Steenberg, Sandgren and Wahren (64,65). However, there seems to be a considerable disagreement about the significance of fines and crill to the structure and properties of the paper made from

[1]Splitting has been omitted since, due to the considerable helical winding of microfibrils in the S_2 - layer of wood pulp fibers, complete splitting of such fibers is highly important.

refined fibers containing fines and crill. A cursory review of the literature indicates that those in favor for a significant role of fines in the structure of paper (66-72) slightly outnumber those of the opposite opinion (15,16,33,65,73,74).

There exists also plenty of experimental evidence for the dissolution of the cell wall material due to refining (26,49-52). The reported amounts of material dissolved in medium to long refining vary from 0.5-4.0% for softwood pulps. More material is generally dissolved from high yield pulps.

Generation of Structural Damage and Modification

This class of refining action is not as clearly defined as the two previous classes. The following subclasses can be separated: (a) cutting of fibers or lamellae[1], (b) generation of axially compressed zones[2] (misalignment zones, dislocations, kink bands), (c) partial cleavage of the cell wall, and (d) creation of invisible weak zones which will lower the cell wall rigidity so that it will collapse[3] locally under drying or so that it will break during deformation[4] associated with tensile or tear loading. There is also considerable evidence about large changes in crystallite sizes and microfibrillar orientation during refining. Since similar changes can also be caused by stresses induced by drying, these structural modifications have been left out as primary effects of beating.

[1]Generation of fines may be a desired refining action in many cases although it takes place through structural damage.
[2]Generation of axially compressed zones may be a desired refining action, for instance, in the manufacture of sack paper.
[3]In many cases the collapse of the lumen is a prerequisite for intense bonding between the fibers.
[4]It should be noted that although refining generates all wall "dislocations," the tensile strength of refined fibers does not decrease but increases (81,93). This only shows the importance of stress equalization between the fibrils and the importance of drying conditions for the tensile strength (86,87). Apparently the dislocation zones are capable of healing under suitable drying conditions.

The direct evidence for fines and crill production has already been cited (see Creation of New Particles). There is also direct evidence for the formation of axially compressed zones in refining (36,44,76-85). It is quite obvious that these misalignment zones play a very significant role during the consolidation of the sheet structure. The mechanisms involved are the Page and Tydeman micro-compression effect (86) and the related Jentzen (87) and negative Jentzen effects (88), which lead to a paper structure where there is plenty of opportunity for dissipation of strain energy (89).

There is no direct quantitative evidence for the partial cleavage of the cell wall due to refining, but in many micrographs of the refined fibers one can observe such cleavages (6,11,12,66). The existence of such cleavages can also be inferred from the cutting of fibers in refining, because it is very probable that not all of the refining action tending to cut the fiber will actually do so.

The collapse of the cell wall lumen due to refining is well accepted (48,90). Many papermakers even believe that collapse of the lumen is a structural prerequisite for strong fiber-to-fiber bonding. However, in the case of certain printing paper grades, the collapsing tendency may be viewed as a structural weakness, because it will cause a drop in the light scattering power of the sheet. Similarly, in the case of linerboard, the collapse of lumen with the simultaneous increased fiber-to-fiber bonding may cause a decrease in the buckling resistance of the linerboard. It has been reported (91) that ultrasonic refining does not weaken the cell wall to the same extent as mechanical refining does, and thus there is less fiber collapse during drying. This can be an advantage in obtaining sheets with higher bulk and tearing strength at a medium level of tensile strength.

One can consider that cutting and partial cleavage form one end of a "damage distribution" curve caused by a certain type of refining action on the

fibers. Besides these two visible damages, there will also be some "invisible" weakening of the cell wall that could initiate a final rupture of the sheet in tensile or tear deformation. It is generally agreed that the points of localized damage to the cell wall will show as points of ballooning of the fibers when placed in strong swelling agents (92).

Thus, the last class of primary beating effects "Structural damage" is a heterogeneous class of effects of various magnitude. In some paper grades the "damaging action" of the refiner may be a very desirable beating effect, in some other grades it is totally undesirable.

Due to the basic structure of the chemical pulp fibers and due to the fact that refining is carried out in water, it is impossible to control the refining so that one obtains specifically only one of the listed primary beating effects (32). It is also easy to accept the view expressed by Steenberg (4) that it is impossible to characterize the beating result by only one single parameter.

HYPOTHESES FOR MECHANICS OF REFINING

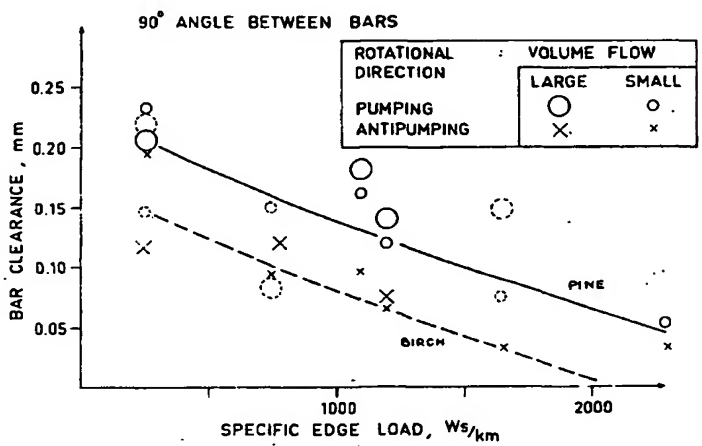
As was already stated, we know very little of the actual action inside the refiner and how this refiner action changes with refining conditions, i.e., the exact transfer mechanics of energy into the fibers are still unknown. In the following, a review of the various hypotheses and theories put forward for describing the mechanism by which fibers interact with the refiner is given. Before doing so, a short summing-up of the present facts of refining are presented, as well as a short description of the current knowledge of the refiner as a piece of hydraulic machinery.

CURRENT FACTS ABOUT THE REFINING PROCESS

All the beating machinery since the invention of the hollander 300 years ago are based on treating the fiber between bars having a relative motion and being loaded against each other in the presence of water. It is well established that, at the consistencies used in conventional beating and refining (2-6%), the fibers do not move independently from each other but instead they form networks, which break and reform continuously. In other words, fibers are present as flocs in low consistency refining.

For refining to take place, the gap between the land areas of the bars has to be small enough, i.e., in the order of a few fiber diameters. It is well established that a water film alone will not support the compressive load exerted on the film between the surfaces of the refiner. When fibers are present in the water, the load carrying capacity (=gap) of the stock depends on the type of fiber (94-97), on pulp drying (95), on shaft power (or loading of the refiner)(94,97,98), on rpm, internal flow field, hydraulic pressure, direction of rotation, and

consistency (95,97,99,113) and on the amount of refining absorbed by the fibers (94-90,100,101,113)(Fig. 3). In this connection it should be noted that the load carrying capacity of a conical refiner and of a hollander, for instance, is quite enormous during refining. When these machines are loaded with pure water, one usually obtains a certain position at the loading device, where the noise generated by the bars is so high that one concludes "there is a bar-to-bar contact." However, during refining one has to go beyond the previous mark quite a lot before the machine starts to pick up load (102,103). The load carrying capacity of the stock to be refined is so high that it actually deforms the refiner or beater (133).



3. Dependence of Bar Clearance on Operational Conditions in a Conical Refiner in the Beginning of Refining (750 rpm)

Although the stock has a load carrying capacity, the land areas in industrial refiners often contact each other in a random manner[1]. This contact will deform the bars; the extent of deformation being dependent on the metallurgical properties of the bars (hardness) and load on the bars (cold hardening). A hypothesis has been proposed, that the wear of refiner plates, which is 40-50 times faster with pure water in between the plates than with 1% stock, is due to abrasion proceeding through minute fissures and impact loadings of these (104). A figure of 20 mg/kWh was given as a specific rate of wear of disc refiner plates.

[1]The contact seems to be more pronounced in the areas where the relative land area in the direction of flow changes suddenly or where there are dams in the grooves of the tackle, or where there are large changes in the orientation of the grooves (130).

The refining results seem to be affected to a large extent by the material of the bars and by the extent of wear (rounding or chisel edge formation) of the edges of the bars (5,95,103-108). A hypothesis has been proposed that plastic refiner plates, due to their softness, will provide a more even distribution of refining results on fibers (109). It has been quite common to recommend a specific metal for a specific refining job, and softer metals were recommended for fibrillating type of refining (110). It has also been shown that, as the refining consistency goes over 10%, the effect of bar edge geometry on refining results practically disappears (111). Refining researchers know how critical the condition and method of conditioning of the bars of the Valley-hollander are for the beating result (112).

THE REFINER AS A PIECE OF TURBOMACHINERY

The conventional bar equipped beater or refiner can be considered as a piece of hydraulic machinery. The simplest case is that of a brake (block, conical, disc). The appropriate formulas have been given, for instance, see reference (114). Results obtained with the brake concept show that when the backed-off idling power correction is done, practically all the refining energy appears as heat (97,115-117)[1].

Dalzell (118) described the power requirements of refining to be composed of two terms (a) fluid film power consumption (brake power, P_1) and (b) pumping and circulation power (idling power, P_2). These two power terms are given in Eq. (1) and (2)

$$P_1 = k_1 \frac{\mu_f}{\Delta^n} DL \cdot \varepsilon \cdot v_{av}^2$$

[1]Theoretical considerations, based on the heat of wetting of the new surfaces generated, could perhaps lead to a prediction that more heat should be recovered as is directed into the refiner in the form of mechanical work.

$$P_2 = k_2 \, DL \cdot v_{av}^{\;3} \tag{2}$$

where

k_1, k_2, and n are constants

μ = viscosity factor (increases greatly with consistency)

Δ = average gap between land areas of rotor and stator

D = effective diameter

L = effective "bar" length

ε = contact area ratio (of the total available area)

v_{av} = effective peripheral speed

According to the author, the disc refiner can have the narrowest grooves between the bars because it is easier to convey the pulp in this type of refiner due to the high centrifugal effect. Dalzell also indicates that, above an efficient peripheral velocity of 25 m/s, the idling power is doing some useful work on the fibers.

Banks (119) maintains that the total power requirement of a refiner is made of three components: (a) power losses due to the whirling of disc in the stock (generation of turbulence in the backed-off position), (b) power losses due to the pumping effect (in unloaded position), and (c) power absorbed in useful attrition work. He gives the following formulas for these three components of refining:

$$P_1 = k_1 D^5 \omega^3 \tag{3}$$

$$P_2 = k_2 D^2 \, \omega^2 \tag{4}$$

$$P_3 = k_3 f F_t D\omega \tag{5}$$

where

k_1, k_2, k_3 are constants

D = effective disc diameter

ω = angular velocity

f = effective friction coefficient

F_t = axial thrust of the rotating shaft

Banks also notes that due to the high centrifugal action helping the transport of fibers, disc refiners can have smaller tackle elements than the fillings of a conical refiner or a hollander. Based on experience he also gives the following engineering guide values for the effective peripheral speeds of various types of refining operation:

(a) 20 m/s, when the predominant characteristic is fiber length control

(b) 25 m/s, when one wants to obtain a balance between fibrillation and fiber length control

(c) 30 m/s, when the predominant characteristic is fibrillation

(d) 35 m/s, when good deflaking or defibration characteristics are wanted

For high energy efficiency the lowest possible speed should be selected. Similarly, based on experience, it is stated that the bar widths should be around 3 mm for case (a) and wider, about 5-6 mm, for case (c). The grooves should be sufficiently narrow to provide an efficient refining action but wide enough so that the stock will pass through; a value of 3-5 mm is suggested as a practical value provided the stock is well fibrillated. A groove depth of 6 mm is recommended as a compromise for good throughput and efficient refining action (proximity factor). A minimum consistency of 3% is advocated in order to maintain the fiber film even at high specific loading values.

Herbert and Marsh (120) give in essence a similar breakdown for the total horsepower requirements of the refiner, as did Banks (119). However, the formula for the power requirement for work absorption is different.

$$P_3 = K_d \, D_i \, (D_o^2 - D_i^2) \qquad (6)$$

where

K_d = disc friction constant, which includes the coefficient of friction between fibers and plate and between fibers themselves as well as the average pressure between the plate "contacting" surfaces

D_i = inner diameter of the disc

D_o = outer diameter of the disc

ω = angular velocity

Based on the formula of Eq. (6), the authors claim that the present disc refiners do not have an optimal ratio D_i/D_o (should be = 0.6) but operate at a too small ratio and thus are less energy effficient. With the proposed improvements, in the ratio D_i/D_o, the authors claim that the energy efficiency of a refiner could be raised from 67% to 72%. The effect of the disc grooves was shown to be a tripling of the hydraulic losses due to turbulence.

Pashinskii (121) has derived a modified Bernoulli-equation for the internal flow phenomena of a refiner. His derivations predict a reverse flow in the stator grooves of the refiner and a mixing flow between the rotor and stator grooves. This phenomenon was actually observed in mill scale refiner trials by Halme and Syrjänen (20,122). Reverse flow has also been reported by Herbert and Marsh (120). Pashinskii suggests that the number of zones, where intermixing of stock between rotor and stator grooves takes place, should be maximized in order to achieve an efficient refining.

Leider and Nissan (117) have considered the power requirement for the actual refining process (total power - idling power) for two hypothetical cases. In the first case it is assumed that the pulp suspension behaves like a solid, i.e., it dissipates energy by disc friction and heats up. In the second case it is assumed that the pulp suspension in between the rotor and stator land areas behaves like a fluid, i.e., it dissipates energy through viscous phenomena in the presence of a shear field.

For the solid continuum case, the net power requirement of refining is related to the operational parameters as follows:

$$P_{net,s} = k_1 \varepsilon^2 \omega f \overline{P}_p (D_o^3 - D_i^3) \tag{7}$$

where

k_1 = constant = $\frac{\pi^2}{6}$

ε = fraction of refining area filled with "lands"

ω = angular velocity

f = effective coefficient of friction for fiber-disc combination

$\overline{P}_p$ = average plate pressure over the involved surfaces

D_o = outer diameter of refining zone

D_i = inner diameter of refining zone

Treating the fiber suspension as a homogenous fluid yields

$$P_{net,f} = k_2 m \varepsilon^2 \omega^{n+1} \frac{1}{(n+1)^n} (D_o^{n+3} - D_i^{n+3}) \tag{8}$$

where

m = viscosity of pulp suspension in the case of laminar flow field, and in the case of a turbulent flow field

= (density)·(Prandl's mixing length)^2

n = 1 for laminar flow field, and

= 2 for turbulent flow field

Δ = gap between the rotor and stator land areas

One can conclude from the previous review that the net refining power requirements are related either to solid friction or to fluid friction between the opposing land areas of the rotor and stator tackle. In the first case the axial thrust appears as the driving force for the energy transfer from the tackles to the medium to be refined and in the latter case the controlling factor is the gap between the tackles. The gap and the axial thrust, however, are not independent, since the gap decreases as the axial thrust to the rotating shaft is increased.

It is most probable that both direct disc friction (metal to fiber and metal to metal) as well as viscous dissipation will absorb the net refining power. This is backed up by the observation that pure water will soon heat up if moderate refining power is used, but at the same time pure water will not support the actual refining power, i.e., the plates will clash (117). But if fibers are present in the fluid, a high refiner load can be applied, indicating a friction phenomenon between the land areas in close contact to each other (see Current Facts about Refining Process, "gap during refining").

Recent studies have shown that the hydraulic pressure pulses inside a refiner are very small. In one case values between 0.003 and 0.01 bar (123) and in the other case values between 0.1 to 0.5 bar (97) were reported. These values seem

to be so small that a direct refining effect by such hydraulic pulses is highly improbable. On the other hand, direct visual observations have shown that cavitation quite often occurs close to the trailing edges of the rotor bars (124,125). It has even been claimed that by increasing the occurrence of cavitation, the efficiency and speed of refining can be increased (126). The large scale occurrence of cavitation (125) may well explain the common recommendation of modern refiner equipment manufacturers, i.e., the refiner must be placed so that it operates under considerable hydraulic pressure.

The cited studies, where the refiner has been analyzed as a piece of turbomachinery, seem to indicate that direct bar-to-fiber contact and intense fiber-to-fiber contact are involved in the refining process. These studies, however, leave open what is the exact mode (or modes) of transferring the energy impacts into the cell wall. In other words, they do not clarify the actual mechanics of the refining action, i.e., is it, for instance, mainly through the action of the leading edge of the bars or through a crushing and bruising action between the opposing land areas of the rotor and stator bars. Besides, the turbomachinery studies by no means describe the transport phenomena of fibers to the position of achieving the refining action, since such studies do not tell anything about the path of average fiber through the beating machine and how the operational parameters affect this path.

MECHANISMS FOR ABSORPTION OF REFINING ACTION INTO FIBERS

Many hypotheses have been put forward during the years to account for the various modes of the absorption of refining work by the fibers. The historical presentations of the action of the beater to individual fibers were deduced from the effects produced on individual fibers. Usually only two types of work transfer mechanisms were accounted for: namely (a) cutting of fibers between closing bar

edges and (b) brushing, crushing, bruising, and/or rubbing of fibers between the opposing land areas of stator and rotor bars (127,128). A large number of formulae have been developed over the years to characterize the cutting action or the fibrillating action of the beater. These formulae have been summarized by Halme (129). One may conclude the historical phase of refiner action on fibers by the following (127):

1. Using a light concentration of stock in the beater with sharp tackle of hard metal and bringing the roll down hard, one obtains quickly a "free" (fast-draining) stock, where the fibers are chopped and cut up.

2. Conversely, with the same furnish but of a thicker consistency and using a blunt tackle of softer metal, one obtains a "wet" (slow-draining) stock by gradually bringing the roll down to a fairly hard rub over a period of several hours.

The time of refining *per se* was not considered to be a criterion of quality.

The classification of the present theories or hypotheses for the transfer of the refining action into the fibers may be done as shown in Table II. This classification is somewhat arbitrary. Many of the listed hypotheses cover only a small area of the refining mechanics and they should perhaps be listed as opinions instead of hypotheses. Along with the introduction of the various hypotheses, experimental evidence in favor or against the proposed mode of transfer of refining action is reviewed.

Fibrage Theory

Smith proposed in 1922 (131) that a beater bar moving through stock collects a beard or fibrage of fibers on the leading edge as does a bedplate bar

TABLE II. PRESENT HYPOTHESES FOR REFINING MECHANISMS

AUTHOR	CLASSIFICATION: NAME	TYPE
SMITH (1923) GONCHAROV (1971)	FIBRAGE	QUANTITATIVE
RANCE (1951) STEENBERG (1951) GONCHAROV (1971)	LUBRICATING SHEAR	QUALITATIVE
DANFORTH (1962) VAN STIPHOUT (1964)	TYPE AND DEGREE OF TREATMENT	QUANTITATIVE
BRECHT AND COWORKERS (1964) SOVIET SCHOOL OF REFINING RESEARCH	INTENSITY/EXTENT SPECIFIC EDGE LOAD/ NET ENERGY CONSUMPTION	SEMIQUANTITATIVE
LEIDER & NISSAN (1977) KLINE (1978)	NUMBER OF IMPACTS/ NET ENERGY PER IMPACT	
HALME (1962) PASHINSKII (1964) FOX AND COWORKERS (1978)	TRANSPORT PHENOMENA I (EMPHASIS ON FLOW BEHAVIOR)	QUALITATIVE
KORDA (1959) ARJAS AND COWORKERS (1969) FOX AND COWORKERS (1979)	TRANSPORT PHENOMENA II (EMPHASIS ON RESIDENCE TIME DISTRIBUTION)	QUALITATIVE
STEENBERG I (1963) GIERTZ (1964) CLARK (1977	DESCRIPTIVE CONSIDERATIONS	QUALITATIVE
STEENBERG II (1979)	(OOZING/CONSOLIDATION)	
PAGE AND COWORKERS (1962) BANKS (1967) EBELING (1979)	TREATMENT OF FLOCS	QUALITATIVE

with stock travelling over it. The beating action is assumed to be caused (a) by the moving bars shearing through the fibrage (cutting action) when a pair of bar edges cross over each other, or (b) by the bar and fibers on the bar edge sliding under pressure over the pile of fibers forming the fibrage on the second bar edge (wet beating action)(Fig. 4). Cutting does not even require the bars to come in contact since shearing effect on compressed fibers is thought to be sufficient to give a cutting action.

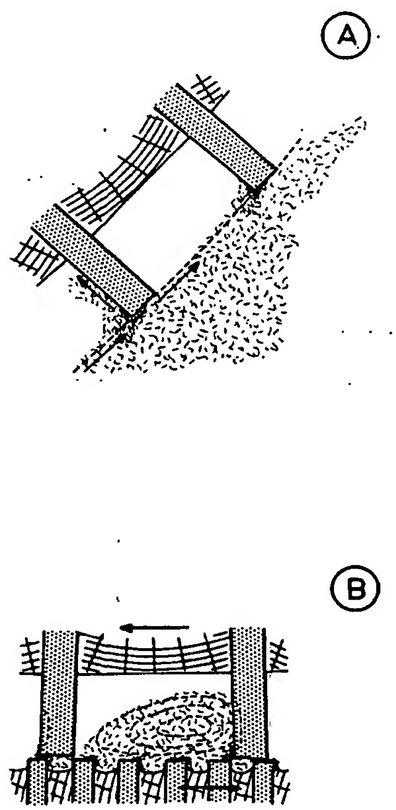


Figure 4. Formation of Fibrages on the Flybars (A) and Bedplate Bars (B) in a Beater (Smith, 1923)

Smith considered the fact that (a) the wear of the front surface of the flybars occurred 2 mm above the leading edges, when beating pigment-containing stock and the fact that (b) when a narrow rod with a square cross section was moved by

hand through the stock, the rod collected fibrages and (c) that on an accidental breakdown of a Jordan refiner, fibrage was observed on the rotor bar edges as evidence for his fibrage theory of refining.

Based on the fibrage hypothesis, Smith also derived formulae for the capacity of the beater and for controlling the type of the refining action on fibers. Since the load of the beater roll is supported by the fibrages, i.e., by the flybar edges, Smith introduced the term "actual beating pressure" (force per fibrage area) to characterize the nature of beating in addition to the consistency of the stock as a means of controlling the cutting action.

In the case of fairly wide flybars and bedplate bars, the actual beating pressure, P, is related to the specific edge pressure P_k (force per length) and the average fiber length l_f, as follows:

$$P \sim \frac{P_k}{\frac{1}{2} l_f} \sim \frac{F_{roll}}{L_{act} \frac{1}{2} l_f} \sim \frac{P_{net}}{L_s \frac{1}{2} l_f} = k \frac{B_s}{\frac{1}{2} l_f} \tag{9}$$

where

F_{roll} = force exerted by the flybar roll against the bedplate

P_{net} = power required to turn the flybar roll in excess of idling power

L_s = cutting length per second

B_s = specific edge load (energy per active cutting length)

k = proportionality constant

Taking into consideration that there is a fairly linear relationship between the edge pressure (force per active cutting length) as defined by Smith and the net power of the beater, one may conclude that the actual beating pressure of Smith is the "forerunner" of the present specific edge load concept.

In other words, assuming that there is a maximum pressure supported by the cell wall, Eq. (9) partially explains why hardwood pulps require considerably less specific edge load than softwood pulps, and why the load carrying capacity of fibers to be refined diminishes as the refining continues (96).

Smith's fibrage theory (131) assumes that the crossing edges of the bars are uniformly covered with fibers. Figure 5 shows values for the fibrage coverage as reported by Smith[1] based on swinging by hand a square cross section rod with a speed of 9 to 10 m/s through the stock. As can be seen from Fig. 5, the average amount of fibrage varies greatly with consistency and with average fiber length, ranging from 0.5 g/m for short fibers at low consistency to 6 g/m for longer fibers at higher consistency. Smith reports (131) that at higher consistencies the fibrage coverage tended to be irregular unless the rod was moved at higher speeds. The amount of fibers deposited increased rapidly with speed.

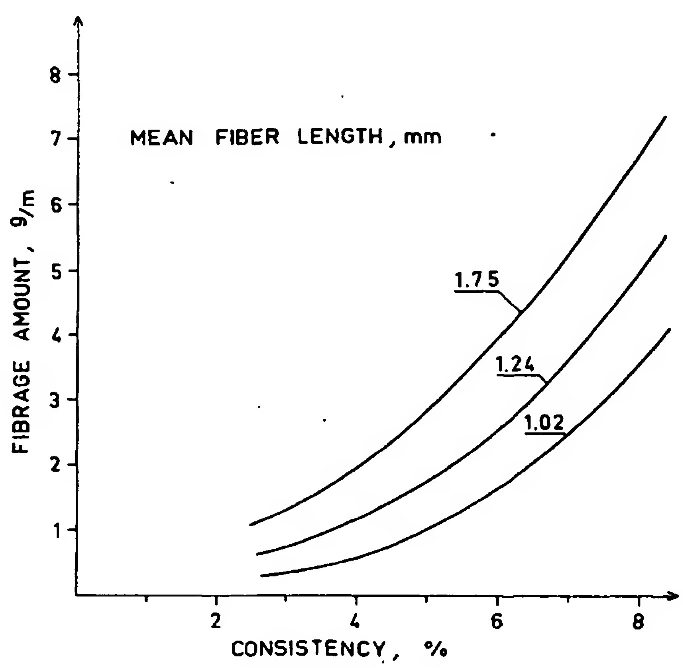


Figure 5. Amount of Fibrage Formed on the Leading Edge of Rod Moving Through Stock (Smith, 1923)

[1]The type of furnish is not reported, but there are indications that it could have been bleached softwood sulfite.

Stephansen (5) has experimented with formation of fibrages under ideal model study conditions. Figure 6 shows his result for bleached sulfite softwood pulp at 3% consistency. The fibrage coverage obtained is of the same order of magnitude as Smith's results. Stephansen reported a value of 3-4 g/m at 10% consistency and with 2 cm approach distance and 5 m/s approach velocity. There is, however, a contradiction between Stephansen's and Smith's results. Smith reported an increase in the fibrage coverage with speed while Stephansen observed just the opposite (Fig. 6). Based on the mass flow through industrial size refiners and on their cutting length per time unit, Stephansen estimated that, on an average, the fiber has a probability to be in a fibrage position about 8 times during its passage through the refiner. This, according to Stephansen, means that a considerable amount of fibers will be treated during the passage. Stephansen also reported that the consistency of deposited fibrages increased with speed, i.e., for 3% stock the consistency of fibrages was about 6% at 1 m/s approach velocity and about 12% at 16 m/s approach velocity. No word was mentioned about the regularity of the deposited fibrages.

Recently Maslakov (132) has analyzed the refining process from a fibrage formation viewpoint. He concluded that the amount of fibrages for an unbeaten pulp was around 4-4.5 g/m for a wide variety of disc refiners and softwood pulps. For a highly refined pulp the corresponding amount was predicted to be 0.5-0.6 g/m, and it corresponded to a monolayer of tightly packed fibers on the bar edges. Maslakov speculated that the sliding of the refined "fibrage" fibers over the bar edges due to their short length and slimy appearance greatly reduced the load carrying capacity of the refiner.

Based on a study of the compressive and shearing forces exerted on the bars during refining, Goncharov (134) proposed a fibrage mechanism to account for the

results obtained. The local compressive stresses measured were 13 times greater than the average pressure calculated by dividing the axial thrust with the average surface area of "contact" between the rotor and stator bars. Besides, the high compressive stress could only be registered at the leading edge of the bar. The width of this zone was 2.5 to 3 mm and it was independent of the width of the stator bars or rotor bars. Goncharov also claimed that the existence of fibrages could be seen in highspeed movies.

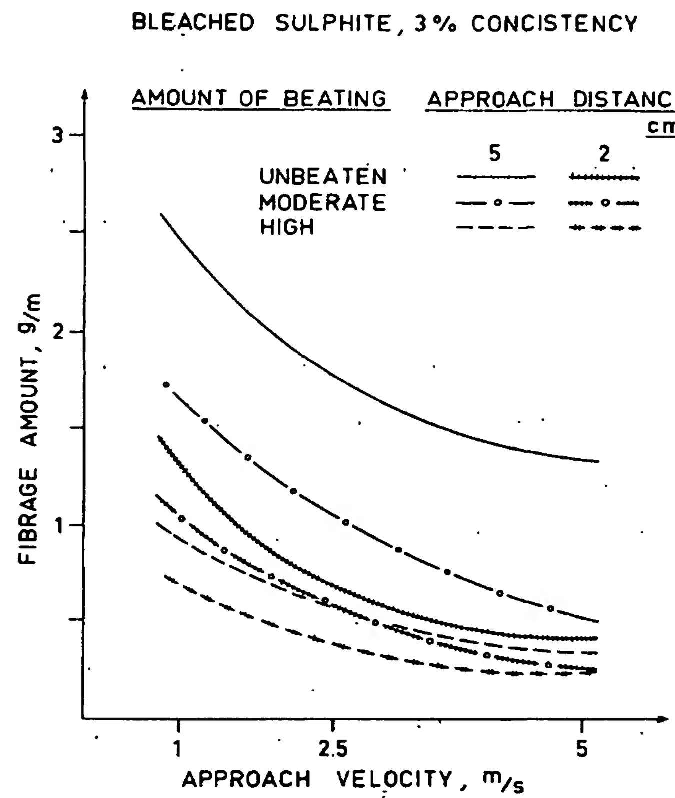


Figure 6. Amount of Fibers Collected on Bar Edge During Model Studies (Stephansen, 1967)

On the other hand, based on high-speed movies of a pilot conical refiner, Page and coworkers (124) reported no homogeneous existence of fibrages at the leading edges of the rotor bars. The same conclusion can be made from the high-speed movie of Halme and Syrjänen (122).

Thus, one has to conclude that, although the fibrage theory of Smith (131) is the only quantitative treatment of the refining mechanics, there is no conclusive experimental evidence available to support the formation of homogeneous fibrage coverage on the loading edges of crossing land areas during LC-refining.

Beating as Lubrication Process

Rance (133) and Steenberg (100) published results in 1951 where the refining had been analyzed as a lubrication process. Rance studied the lubrication behavior of a high-speed refiner and of a Jordan while Steenberg used a Valley hollander in his studies.

According to Rance (133), in the case of a high-speed refiner, the shell setting curves offered indirect evidence of the occurrence of (a) fluid (hydrodynamic) lubrication, (b) boundary lubrication, and (c) lubrication breakdown, which ended up into excessive metal wear or sizing up of the refiner. It was stated that the most economic refining should be done at loads near the breakdown of the "lubricating fiber film."

The hydrafiner was depicted to be operating under such conditions, i.e., at loads near the lubrication breakdown conditions, and the refining action under these conditions involved a high degree of surface fibrillation with some fiber shortening due to shearing. The Jordan instead was visualized to be working under conditions of lubrication breakdown, i.e., with pressure and peripheral speed such that a stable lubricant film could not be maintained. According to Rance, a pulp that

exhibited a rapid gap reduction in a Valley beater test was a low quality pulp for industrial refining under boundary lubrication conditions. Similarly it was speculated that the reduction of load carrying capacity during beating was related to the accompanying reduction of the average fiber length.

Rance, however, warned about drawing too many conclusions from the lubrication theory to refining, since in beating one wants to alter the lubricant, i.e., change the character of the fiber, while in lubrication the quality of the oil should remain unaltered.

Steenberg (100) carried out his experiments with a Valley beater varying the consistency, load and peripheral speed. Figure 7a shows the variation of the apparent friction coefficient (μ) with load (F), consistency, (c), and time (t). Figure 7b shows the dependence between the coefficient of friction and the consistency at various loads during initial beating, and Fig. 7c shows the decrease of the apparent viscosity of the pulp during beating based on "force fitting" the various time curves of Fig. 7a into a master curve shown. The pulp used in the experiments was a hot alkali treated dissolving pulp, i.e., a pulp of extremely slow beating. Steenberg stated that the fall of the viscosity would be considerably larger in the case of fast beating pulp.

Based on results in Fig. 7 one may conclude that Valley beating is carried out under hydrodynamic (fluid) lubrication conditions, and that the work absorption capacity, i.e., coefficient of friction, decreases with beating as does the apparent viscosity of the pulp. Steenberg also showed that the gap holding capacity of a pulp during refining is not solely dependent on the average fiber length, but irreversibly related to the slime formation tendency of the pulp.

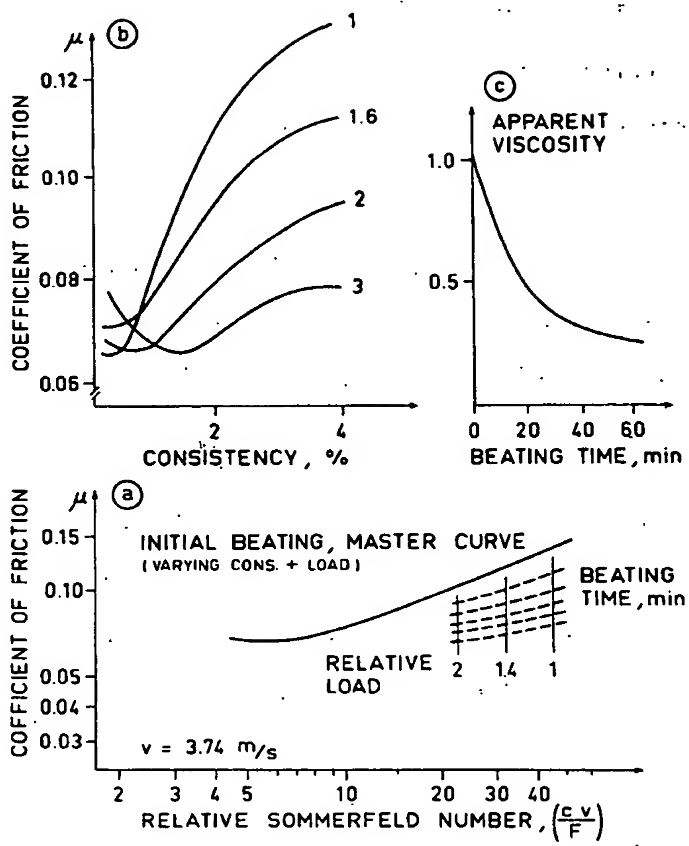


Figure 7. Apparent Viscosity and Coefficient of Friction of Dissolving Pulp During Beating (Steenberg, 1951)

The reported values for coefficient of friction by Steenberg vary between 0.06 and 0.14; higher consistencies and lower loads giving larger values. Goncharov (134) reported a value of 0.11 for the coefficient of friction of refining sulfite pulp in an industrial disc refiner at a consistency of 2.5 to 3%. This value was obtained by dividing the tangential force measured with a strain gage device on the stator bar with a simultaneously measured normal force. The value of the friction coefficient decreased towards the periphery. When the average coefficient of friction is estimated from the net turning power and from the axial thrust given in Goncharov's article, one obtains a value of μ = 0.05-0.08. This difference between the locally measured coefficient and coefficient calculated from the overall force

and power balance seems to indicate that the lubrication phenomena during refining are highly local.

Figure 8 shows the results of Goncharov's measurements (134). Figure 8a applies to a case where the distance between the land areas of the rotor and stator bars is large (0.15-0.3 mm) and the possible fibrage coverage very small. In this case the maximum calculated pressure against the land area of the bar edge was below 2 MPa (20 atm.). In Fig. 8b the maximum calculated pressure is around 3.5 MPa (35 atm.) and it corresponds to a case where the specific edge load is 1.5 Ws/m of cutting length. In this case the specific compressive stress is practically constant for a penetration distance of 2.5 to 3 mm between the bars (distance a in Fig. 8b). Goncharov explains this by the squeezing of two fibrages into the advancing gap (Case 1 in 8c). This causes the phase of the maximum compression stresses. It is at this stage, claims Goncharov, that the most intensive refining action takes place through carding and crushing. If the fibers are placed under too high shear stresses at this stage or during the following separation phase (Case 2 in 8c), they will break in tension[1]. During the phase 2, the pressure quickly decreases (distance b in Fig. 8b) to a level which is only about 10 to 15% of the maximum pressure.

One could perhaps advance the explanation given by Goncharov by speculating that if the fibers really enter into the advancing gap as thick fibrages, the compressive stresses that these shearing fibers will support are large enough to cause tremendous movement of water inside the cell wall and aid in creating internal fibrillation and external fibrillation. The latter effect of refining is, of course, greatly accelerated by the mutual rubbing of the fibers.

[1]Steenberg (4) has speculated that the shortening of the fibers during refining is through tension failure and not through cutting, since the ends of broken fibers become highly fibrillated

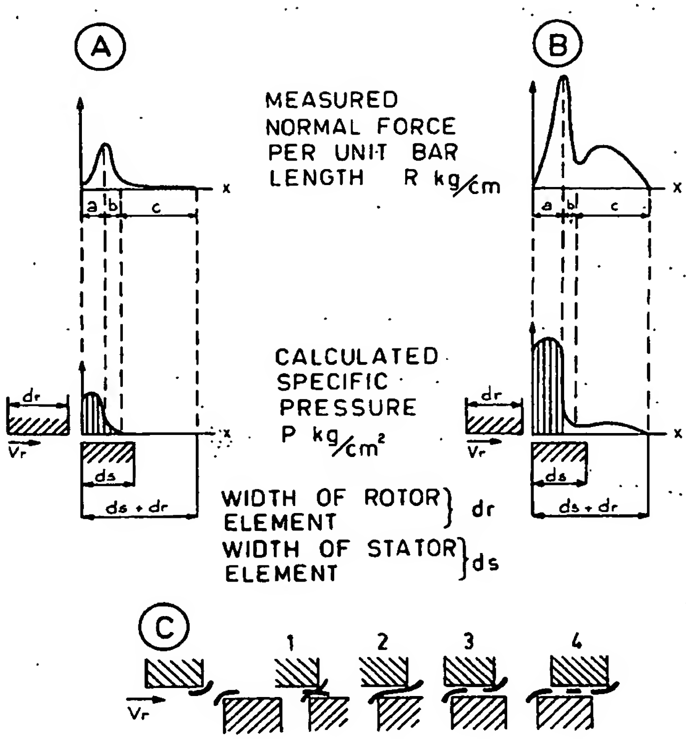


Figure 8. Fibrage Induced Bar Forces (Goncharov, 1971)

Type and Degree of Treatment Concept

In this theory, refining is actually considered as a black box operation which can be controlled by an intensity term and by an extent term. The theory usually bears a name: "Specific Edge Load Theory of Refining," and it is usually credited to Brecht and coworkers (135,138) who advanced the specific edge load idea of Wultsch and Flucher (98).

However, the idea of a certain type of specific edge load had already been used by Smith in 1951 (139) in trying to explain differences in the quality of hollander and refiner beaten stock. Moreover, Cottrall (103) states the significance of increased edge loading as: "At higher loads, the fiber film between the bars gets thinner. This has two effects - (1) less fibers get between the bars in each pass, so that less fibers are treated at each pass and the proportion of

untreated fibers is greater - (2) the fibers that are treated are more intensively treated ...."

In connection with the specific edge load theory, one should remember that Lewis and Danforth (140) proposed already in 1962 that the stock preparation process should be expressed as a function of two independent components, namely (a) number of impacts between the tackle edges to which the fibers are subjected and (b) the severity of such impacts (Fig. 9). This idea was later developed to a quantitative characterization of refining by Danforth (141). Also one should remember the pioneering work of Van Stiphout (142) in characterizing the nature and extent of the refining conditions. Van Stiphout apparently was also the first to use the concept of plotting the dependence of the refining result as constant value curves (isocurves) in the amount of refining and quality of refining coordinate system. Van Stiphout concluded from his result that refiners of greatly different size, tackle, and rpm can be made to treat the pulp in quite the same manner, provided that the two components for the characterization of the refining conditions are about the same. He also proposed that the refiner for a paper machine, with a variety of paper grades, should be equipped with a variable speed transmission. The same idea has later been advocated by Arjas (143).

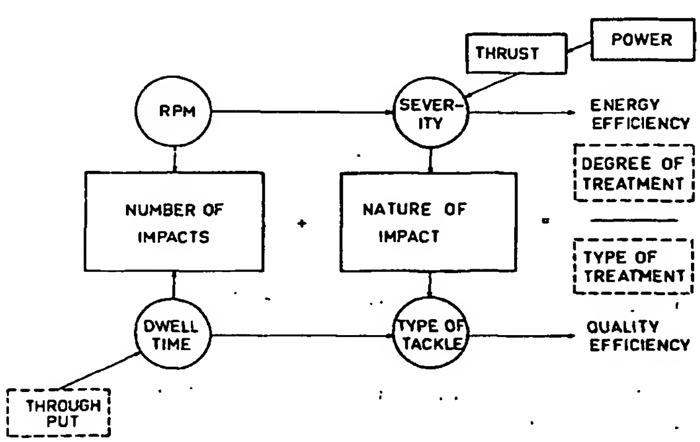


Figure 9. Basis for Analysis of Stock Preparation (Lewis and Danforth, 1962)

Leider and Nissan (117) have also derived equations to characterize the number of impacts and energy released per impact. Some criticism has been directed towards the assumptions under which the equations were derived (151). Lately Kline (152) has proposed that the intensity of refining should be characterized by net horsepower applied divided by effective (average) refining area, and the amount of refining should be characterized by a term related to the quotient of the effective (average) refining area divided by the mass flow of pulp. The product of the intensity and amount terms will give the specific net energy consumption of beating.

The specific edge load theory is based on a tacit assumption that the major part of the refining action is due to the deformation induced by the impact of opposing rotor and stator bars as they cross over each other. This impact phenomenon is directly related to the "cutting speed" L_s (edge length per second, inch cuts per second). The power consumption of the refiner, P_{total}, is divided into two components, P_{idle}[1] describing the power with the rotor in the backed-off position and water flowing through the refiner, and P_{net}, which is the difference between the total power consumption and the idling power. The intensity of refining is defined by the term specific edge load, B_s, which is

$$B_s = \frac{P_{total} - P_{idle}}{L_s} = \frac{P_{net}}{Nl_s} \qquad (10)$$

where

$L_s = Nl_s$, i.e., rps times the total cutting length of the refiner in question.

[1]Steenberg (167) has criticized this principle, since the fibers suspended in the water change the viscous drag of pure water considerably.

The amount of refining is defined as "specific net energy consumption," W_e and it is

$$W_e = \frac{P_{total} - P_{idle}}{\dot{m}} = \frac{P_{net}}{\dot{m}} \tag{11}$$

where

$\dot{m}$ = the mass flow of pulp (dry) through the refiner.

Based on an extensive series of experiments, Brecht and Siewert (136-137) concluded that the refining result of a given pulp is unambiguously defined when the B_s and W_e of the refining treatment have been the same. In other words, the width of the bars, the number of the bars, their average contact area, rpm, consistency, and volume flow have no other effect on the refining than that included in the definition of the terms B_s and W_e.

Later studies have, however, shown that the specific edge load theory is not a comprehensive system for characterizing the refining conditions and predicting the refining results. For instance, it does not take into account the effect of the bar material and the sharpness of the leading bar edge on the intensity of refining (see Current Facts about the Refining Process) and it does not take into account the effect of consistency[1] on the intensity factor (144) or the effect of impact angle, direction of rotation, rpm, and depth of the grooves (18,144-147) on the intensity and amount factors of refining. The specific edge load theory has also been criticized because it puts too much emphasis on the impact phenomenon when the opposing bars pass over each other. Note there are plenty of efficient refiners in industrial use where the impact phenomenon - in the sense it is included in the specific edge load theory - is totally missing. For instance, in the Vargo refiner

[1]Both the analysis of Van Stiphout (142) and of Danforth (141) included consistency in the intensity and amount terms.

(148) and in the refiners equipped with basalt tackle (149,150) one may obtain excellent refining results with considerably less energy consumption than in the case of conventional bar filled refiners.

One can conclude that, although the specific edge load theory of refining has greatly clarified the effect of various design parameters of the bar filled refiners on the refining conditions and on the obtained refining action, and although it has properly emphasized the important role of the specific edge load in the intensity factor of refining, it has, perhaps unduly, stressed the scissor-type cutting action in the analysis of refining mechanisms.

Transport Phenomenon in Refining

Mention has already been made of the role of reversing flow in the grooves of the stator (20,119,122) and between the grooves of the rotor and stator tackles (121). These flow patterns define the gross pulp transport through the refiner. The transport phenomenon can be analyzed (a) from the flow behavior viewpoint and (b) from the viewpoint of residence time distribution of pulp fibers inside the refiner.

Flow Behavior

Banks (119) described results of high-speed cinematography of pulp flow through an experimental transparent disc refiner. The quality of the film did not allow a detailed analysis of the flow behavior of fibers inside the refiner. Fibers and flocs were seen to get stapled against the leading edge of the stator bars. The land area of the stator bar covered by these fibers and flocs was about 50 to 70% of the width of the stationary bars[1]. The flocs remained on the stator bar for at

[1]No information was given about the consistency of the pulp or the width of the stator bars.

least one rotor revolution. More pronounced floc collection was observed towards the disc periphery.

Fox and coworkers (153,154) have reported results of a high-speed cinematographic study of the flow behavior of fibers inside a transparent disc refiner. The studies were carried out with bleached southern kraft pulp using consistencies of 0.1 to 0.3%. Their results are summarized in Fig. 10.

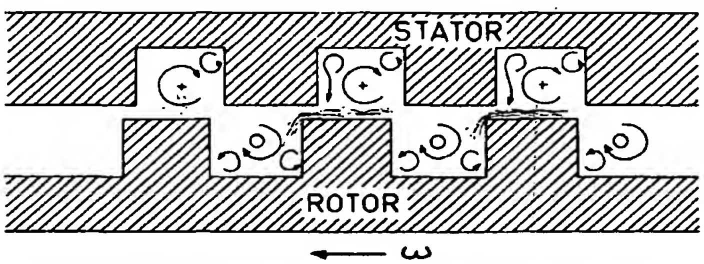


Figure 10. Internal Flow Field and Transport of Fibers Inside a Disc Refiner (Fox, Brodkey, Nissan, 1979)

Fox and coworkers named the various flows as follows:

(a) primary radial flow that is outward in the grooves of the rotor tackle and inward in the grooves of the stator tackle,

(b) secondary vortex in the above mentioned primary flows due to the "spinning" introduced through the friction of the land area of the rotor bar when sliding over the groove of the stator and by the land area of the stator bar when sliding over the rotor groove, and

(c) tertiary flow resulting from the secondary vortex flows in the corners of the grooves. In the case of the stator groove, the vortex flow above the corner of the leading edge of the stator bar is modified into a tertiary wiping flow over the leading edge. The driving force for this flow is the higher pressure that exists in the stator groove.

Fox and coworkers (153,154) proposed that this tertiary flow empties fibers from the stator grooves into the refining gap and holds them against the leading edge of the rotor bars. Stapling of the fibers was not observed against the leading edge of the stator bars in the high speed movies except at the periphery of the stator tackle. The authors hypothesized that it is the stapled fibers that receive the refining action and after breaking loose they either become part of the inward stator flow and perhaps get stapled again or they become part of the outward rotor flow and may leave the refiner or be reverted back to the stator through the outside annulus of the refiner. In a later paper Fox (154) proposed that high levels of fluid and mechanical shear act to cut and refine the stapled stock and that three modes of delivery are involved in delivering the stapled fibers to the periphery. These proposed modes are: (a) release delivery, (b) slip delivery, and (c) sweep delivery. The last mode is pictured to take place only in the so called exit-flow region, i.e., in those rotor and stator grooves which connect the inlet flow region to the exit region. Fox also proposed that there is an optimum angular velocity at which the stapling is maximized. As evidence for this proposal, Fox presented results which indicate that there is a maximum in the thickening effect of the refiner as a function of rpm. In other words, the average consistency of the stock inside the refiner is higher than the consistency of the inlet flow because of the stapling phenomenon.

Since the main work of Fox and coworkers (153,154) was based on extremely low consistencies and since the bar clearance during the test runs was fairly high ($\geq$ 0.15 mm), the results do not necessarily apply to industrial refining, where the bar clearance is considerably smaller and the fibers are not able to move independently because of the network restrictions originating from the higher consistency of the stock. Besides, there is a discrepancy between the results of Banks (119),

who observed stapling against stator bar edges, and those of Fox et al., where stapling was observed mainly against the rotor bars.

Residence Time Distribution

Ryti, Arjas and coworkers (101,155-161) have studied in considerable detail the role of residence time distribution in refining. The starting point of the study was that the refining action is related to the residence time inside the refiner and to the probability of treatment during this time. In other words, their starting point follows the earlier idea of Steenberg (4), who stated that the refining action is a result of a selection process and of a treatment process. In practical studies, Ryti and Arjas concentrated only on the analysis of the residence time, discarding the contribution of the probability function. The tacit assumption in their studies was that the beating result is better the more uniform the refining treatment has been[1].

In the preliminary experiments it was observed that systematic differences existed between the drainage properties of the stock and the physical properties of the handsheets when continuous refining in the Valley beater was compared, at equal effective refining time, with the results obtained from normal, "batch type" Valley refining (101,155). The differences were not statistically significant. For instance, when tear was plotted as a function of tensile or scattering power as a function of tensile no significant difference was observed between the two sets of refining in the Valley beater, i.e., the type of refining action produced by the continuous and by the batch operation of the Valley beater did not differ significantly. However, the authors concluded that the results of the preliminary

[1]The practical application of this assumption may be questioned, since in most cases of industrial paper production it has been found that significant economical and property advantages can be obtained by using a mixture of various types of pulp fibers as a raw material for paper.

experiments support the hypothesis that the shape of the residence time distribution curve affects the papermaking properties of the pulp.

In a later study (158) an attempt was made to study the effect of flow rate and angular velocity on the shape of the residence time distribution curve. The residence time curve was measured inside a mill scale conical refiner with conventional pulp fibers tagged with a radioactive chemical. No clear-cut effect of the two studied variables on the RTD-curve could be observed because of very large scatter of the recorded signals. Based on the use of a special levelling technique (157), the authors concluded that the qualitative information was in accordance with the flow behavior one would predict from the role of return flows inside the refiner. Similar information has been observed also in residence time distribution studies of the disc refiner (125), i.e., the flow rate has an effect on the mean residence time, but the effect of the angular speed is not very large. In the last set of experiments (160) the effect of residence time distribution in a mill scale conical refiner on the properties of an unbleached pine kraft pulp was studied. The average residence time was kept constant and comparisons were made between continuous refining (a) in four refiners connected in series, (b) in one refiner, and (c) in one refiner connected with a mixed recirculation tank. The authors concluded that four-fold passage through a refiner produced a more homogeneous refining action than the one-pass refining in accordance with the theory (156). However, again the observed differences in the handsheet properties were not statistically significant. The same result concerning the sharp vs. wide RTD of refining has already been presented by Maynard (102) after studying refining with a high-speed conical refiner equipped with a recirculation valve. Even in the case of refining with recirculation through a mixed chest vs. four-fold or direct passage through the refiner, Arjas and coworkers did not get any significant differences in the beating response (160). Similar results have been reported by Leider (162).

The theoretical treatment of series connected refiners by Arjas (156) gives a different conclusion than the analysis by Korda (163), who came to the conclusion that refiners should be connected in series but equipped with a recirculation flow after each refiner in order to secure the most homogeneous treatment of fibers.

It has been shown that with conical refiners, where the cutting angle between rotor and stator bars differs by about 0°, the residence time distribution curve is sharper (145). It has also been shown (146) that when the mean residence time in the disc refiner decreases while the specific edge load stays constant, the refining becomes more intense, especially in the case where softwood pulps are refined. A statement has also been made that the dwell time inside a disc refiner is only about 1/10 of that in a conical refiner (118).

It thus seems that the average residence time inside the refiner - and especially inside the refining zone[1] - has an effect on the type of refining action experienced by the fibers. However, it seems debatable that refiners connected in series would give a significant advantage in actual refining over parallelly connected refiners. This is especially so if one keeps in mind the flexibility requirement of industrial refining systems. Besides, none of these studies can be used as a guide for how to split the energy of refining between the various refiner units in a series connection case.

It seems to the author that if the role of the residence time distribution curve in refining is to be clarified, one needs to follow the basic analysis of chemical engineering reaction kinetics as, for instance, outlined by Levenspiel for cases with various types of feed-back flow of macrofluids (164). In other words, it is not enough to look only to the residence time distribution. Instead one has to

[1]It should be kept in mind that the actual bar area volume of the refiner is only about 20-25% of the inside volume of the disc refiner and considerably less in conical refiners.

take into consideration also the reaction kinetics, i.e., the treatment function (probability and type of action received).

Descriptive Models

Reference has already been made to the analysis of refining by Steenberg (5). Steenberg stated that several subprocesses, each one having a major influence on the refining result during certain phases of refining, are functioning in refining and therefore a single parameter cannot be used to describe either the process or the state of the product (i.e., beaten pulp). Steenberg advocated that at least two of the subprocesses, namely the selection process and the treatment process, should deserve more attention. He also stated that too much emphasis has been placed in the past on direct tackle-fiber interaction as a mechanism of transferring refining action into fibers and the interaction effect between the fibers has obviously been overlooked. This interaction was visualized to be high enough - because of the network structure of the stock - to cause external fibrillation and crill formation plus increase the flexibility of the fibers and perhaps causing shortening of the fibers through shear-induced tensioning.

Giertz (32) postulated that the intensity of the refining forces acting on fibers can be described by a distribution function (Fig. 11). The problem in industrial refining was depicted to be, that when the intensity of refining was increased in present refiners, the relative role of "unproductive" refining action still stayed at a high value, explaining the low energy efficiency of refining. The same conclusion has been presented by Leider and Nissan (117).

Clark (16) has summarized the mechanics of refining action in a descriptive way. In Clark's treatment one, two or more fibers are depicted between the approaching bars (Fig. 12). A shear force field can accomplish shortening of fibers through shearing tensioning (directly or through the network) and crushing.

Internal and external fibrillation plus production of debris is pictured to take place through abrasion by the bar surfaces and through rolling and twisting action of individual fibers or of several fibers in the bar gap. Dislocations were pictured to form when a floc was being squeezed into the gap between opposing bars.

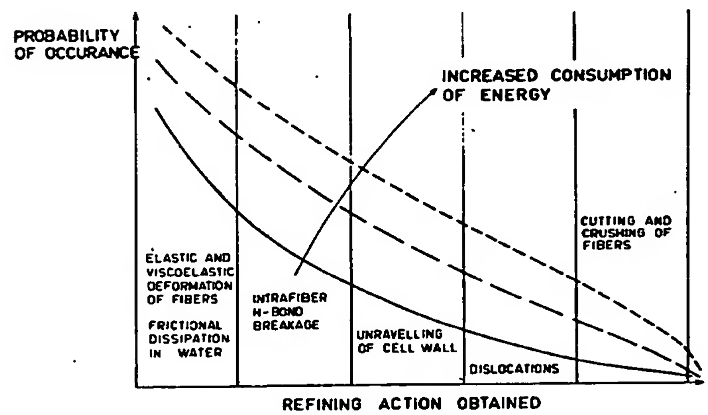


Figure 11. Distribution Curve for Refining Forces (Giertz, 1964)

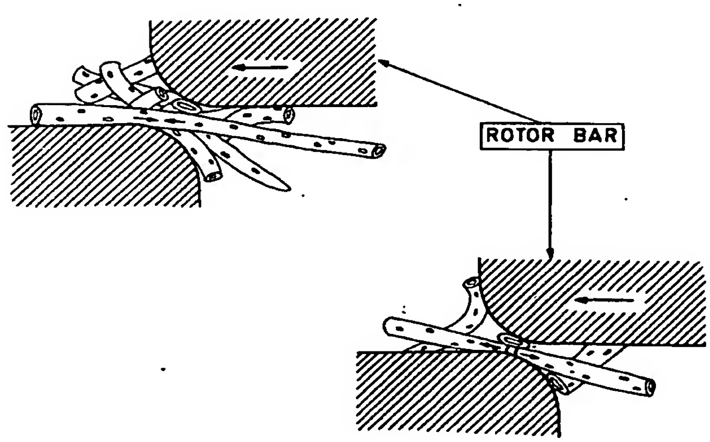


Figure 12. Description Representation of Some Bar Actions in Refining (Clark, 1978)

Steenberg (19,165) has analyzed the refining process as an irreversible kappa process, which involves a critical path for the formation of a stress concentration chain, in which a structural breakdown will occur. For another structural breakdown to occur the particles, i.e., fibers, need to be rearranged.

According to Steenberg there is a threshold consistency below which the formation of a stress concentration chain is impossible because the fiber slurry will "ooze" (i.e., escape the force which is trying to accomplish the stress concentration chain). Above the threshold consistency the network will consolidate under the action of the refining force and a critical path of stress concentration will occur and cause a major primary effect of refining. The threshold consistency will depend, besides on the quality of pulp, on the overall consistency of the stock, on the rate of movement of the force transferring surfaces and on their relative distances, and on the state of beating of the stock. Increased beating will move the threshold consistency towards higher values. The grooves of the refiner play an important role, according to Steenberg. They allow efficient mixing of the pulp fibers to take place after the kappa process and thus generate a new configuration for the next kappa process. A similar idea has also been proposed by Halme and Syrjänen (122). It should also be noted that the oozing/consolidation phenomena takes place in Kollergang refining (166) and that it is impossible to refine in the Kollergang if the overall consistency falls below a certain value.

"Treatment of Flocs" Hypothesis

In the 1951 symposium on beating, Steenberg (100) showed that the gap holding capacity of the Valley-beater could - almost instantaneously - be reduced by adding to the stock a certain amount of slime producing substances (Fig. 13). Based on this observation Steenberg questioned the then accepted idea that the gap is related to the average fiber length of the stock to be refined (133). Later Arjas (101) has reported that when unrefined fibers were quickly replaced into a Valley-beater when the beating was in progress, the instantaneous increase in the gap was related to the amount of stock being replaced, but the decay of the instantaneous gap increase was faster the later in the beating the replacement was done.

During the discussions of the 1951 symposium an idea was proposed that the observed gap effect could be due to dispersion of flocs (168). The highly unstable and erratic gap in the early stages of Valley-hollander beating (169) could afterwards also be considered as a possible indication of flocs being present in the gap.

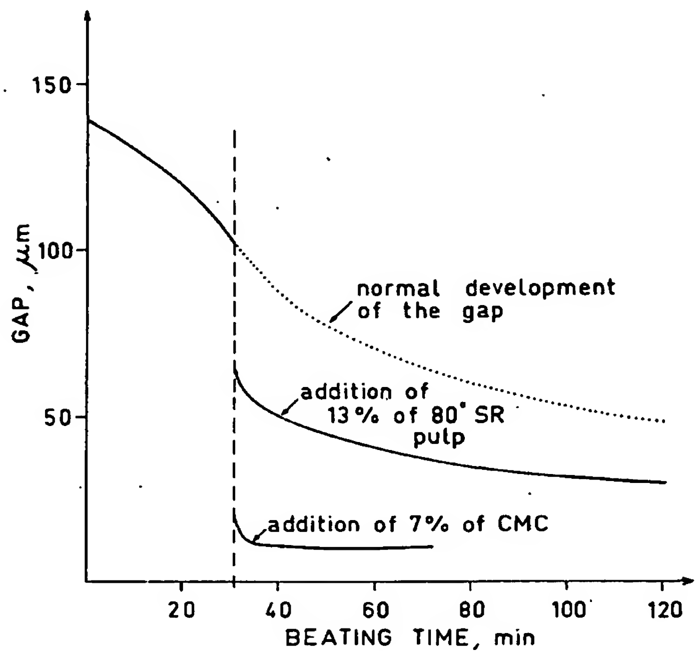


Figure 13. Development of the Refining Gap During Valley-Hollander Beating of Sulfite Pulp (Steenberg, 1951)

The idea that the refining of pulp fibers should actually be considered as refining of mainly flocs did not gain any popularity, although indirect evidence seemed to speak for it. In 1957 Steenberg (170) cited an example that it was impossible to pass fibers through an experimental disc refiner unless it was equipped with semisphere holes linked to each other in succession in rotor and stator discs. He later also reported (171) that it was extremely difficult to measure the viscous shear properties of pulp suspensions in a smooth surfaced plate viscometer because of the presence of fiber bundles and flocs. When narrow radial grooves were machined on the surfaces, an even flow through the measuring zone was obtained.

Page and coworkers (124) apparently were first to actually state that refining involved breakage of flocs and treatment of the remnants of these flocs between the rotor and stator surfaces. They derived their conclusions from high-speed photography of a conical refiner. According to them, the refiner is inefficient since only a small proportion of fibers are where they should be.

In 1967 Banks (119) - based on high-speed cinematography and on contributions by Espenmiller - summarized the mechanics of refining as follows:

1. Flocs consolidate when they are trapped between approaching tackle elements.
2. Mechanical pressure induced by the tackle elements becomes high enough and causes plastic deformation in the fibers composing the floc. Consolidation continues.
3. The floc compressed between the bars is sheared; flocs (and fibers) are ruptured.
4. Release of mechanical pressure allows absorption of water to take place into the ruptured fibrils and fibers.
5. Turbulent agitation may disperse the floc or its remnants into the general mass flow.

According to Banks, the floc treatment theory differs from the fibrage theory in the local action aspect since the fibrage theory assumes a complete uniform coverage of the bar edges. As a matter of fact, the hypothesis of Banks (119) is similar in many respects to that expressed by Page and coworkers (124).

The new and important aspect of this hypothesis for the theory of refining is that fibers are not treated as independent particles, at least in the beginning of refining, but that they take part in refining as entities of macrostructure,

namely as flocs. The probability of a floc getting sheared in between the crossing land areas of the rotor and stator bars is several orders of magnitude smaller than that of an individual fiber. On the other hand, if and when the floc gets entrapped between the bars, the result of refining action may be considered catastrophic for the majority of those fibers which form the floc.

The idea of refining flocs instead of individual fibers is by no means unnatural. It is well established that fibers form strong networks at 2-4% consistency and that, when such networks break up, flocs are formed.

As an additional piece of indirect evidence for treatment of flocs in refining, the noise phenomena during refining should be mentioned. It has been established that the noise level of refining with a hydrafiner goes down very rapidly at the beginning of refining (98). The starting level and speed of decrease was higher for "strong" pulps than for low yield "soft" pulps. The reduction in noise level was most pronounced in the higher harmonics of the "bar contact" frequencies.

NEW EVIDENCE FOR THE HYPOTHESIS OF "TREATMENT OF FLOCS"

Recent high-speed cinematography studies (172) have shown beyond any doubt that the hypothesis of "treatment of flocs" describes the fundamental phenomena in transferring the refining action into the fibers (Fig. 14,15). Movies were taken with a speed of 1000 frames per second. The peripheral speed of the 12 inch disc was gm/s. The film speed was not high enough to stop the rotor bar; it moved about 3 mm during the exposure. A bleached southern pine kraft at 1.1% consistency was used. The gap was about 0.15 mm. The transparent experimental refiner has been described by Fox (153,154). Just before filming, a small amount of black dyed pulp fibers was injected into the eye of the refiner.

The enlarged film frames (Fig. 14,15) show a fairly dark band across the frame. That is the stator groove. The width of this groove is 6 mm. Across this groove are seen the rotor bar edges. The angle between the rotor and stator bar edges is about 20°. In some individual frames the contour of rotor bar edges has been outlined. If there are no fibers between the land areas, then the light from the opposing side of the refiner will pass with ease to the lens of the camera, which is aimed into the light passing through the refiner from the stator side. If there are plenty of fibers between the land areas, the dark flocs will show up as black areas in the crossing of the land areas. If there are plenty of dyed fibers in the rotor groove while it is over the land area of the stator bar, the rotor groove will show up as a light gray band against a white surrounding.

Figures 14 A and B plus 15 A and B show that only occasionally can one find fibrous material between the land areas. Based on an analysis of the film, one could perhaps state that only about once in every ten successive land area crossings is there plenty of fibrous material in between. The shape and optical density of

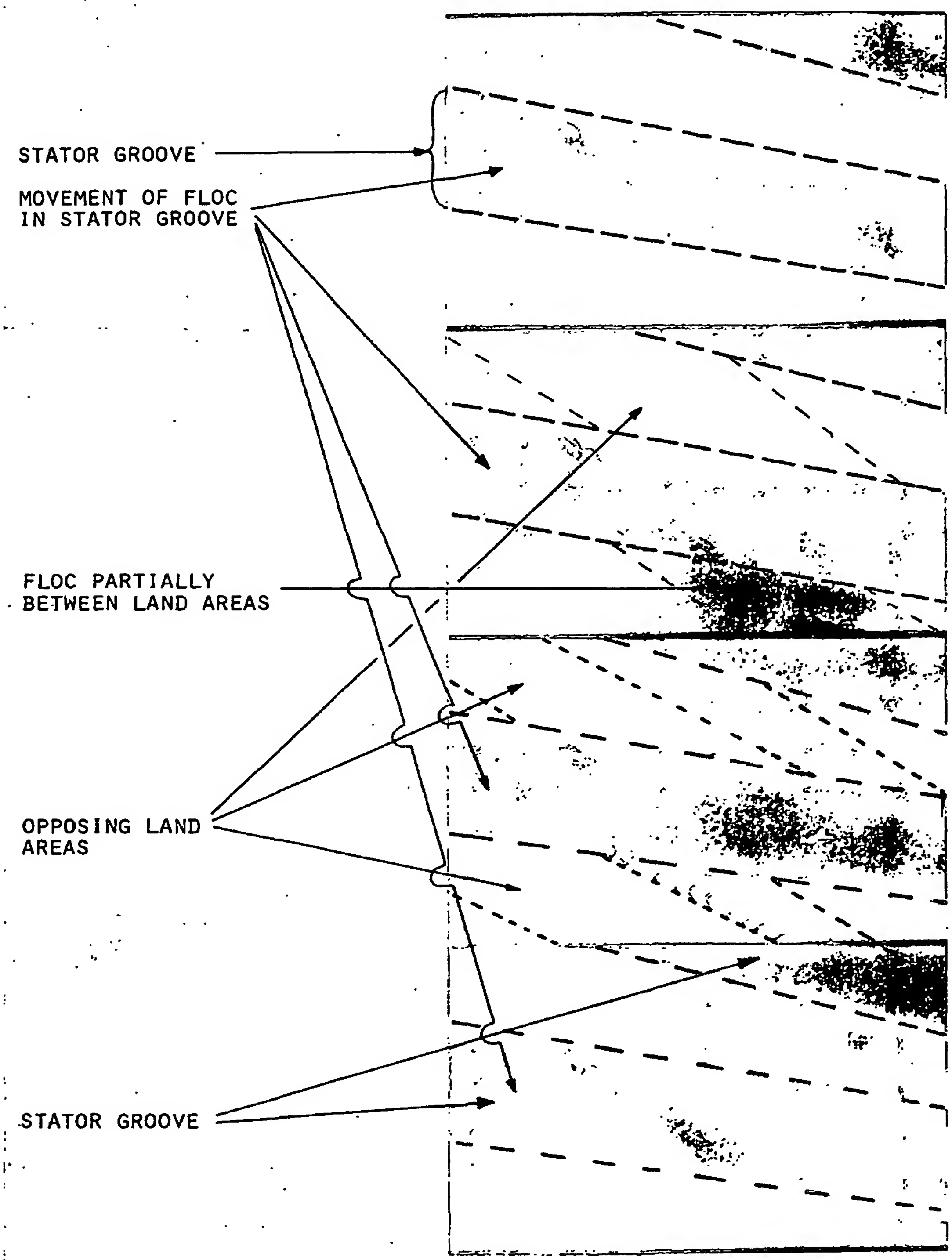


Figure 14. Enlarged 16 mm Film Frames Photographed Through Transparent Experimental Disc Refiner (Successive Frames Exposed at 1/1000 s)

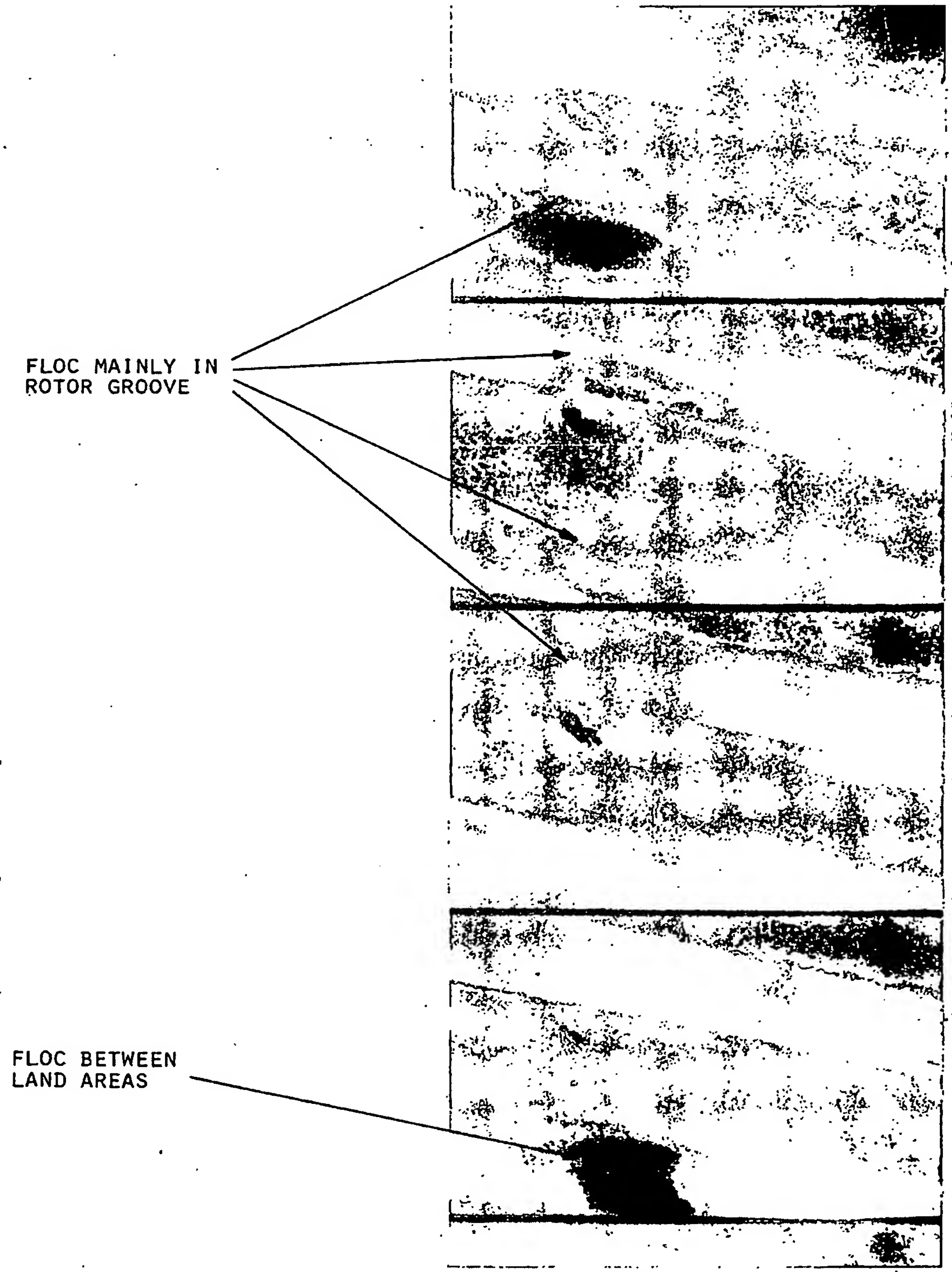


Figure 14. Enlarged 16 mm Film Frames Photographed Through Transparent Experimental Disc Refiner (Successive Frames Exposed at 1/1000 s)

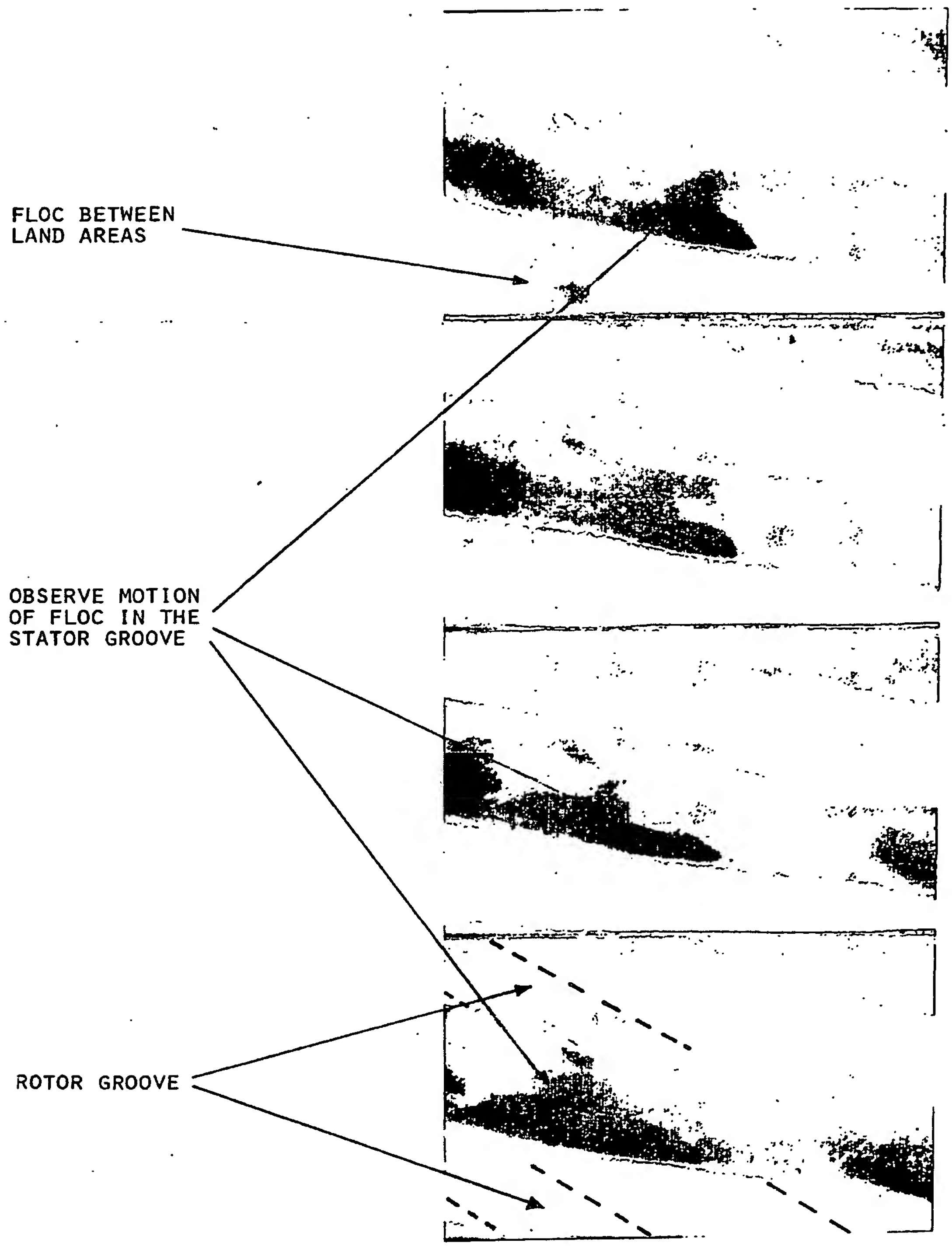


Figure 15. Enlarged 16 mm Film Frames Photographed Through Transparent Experimental Disc Refiner (Successive Frames Exposed at 1/1000 s)

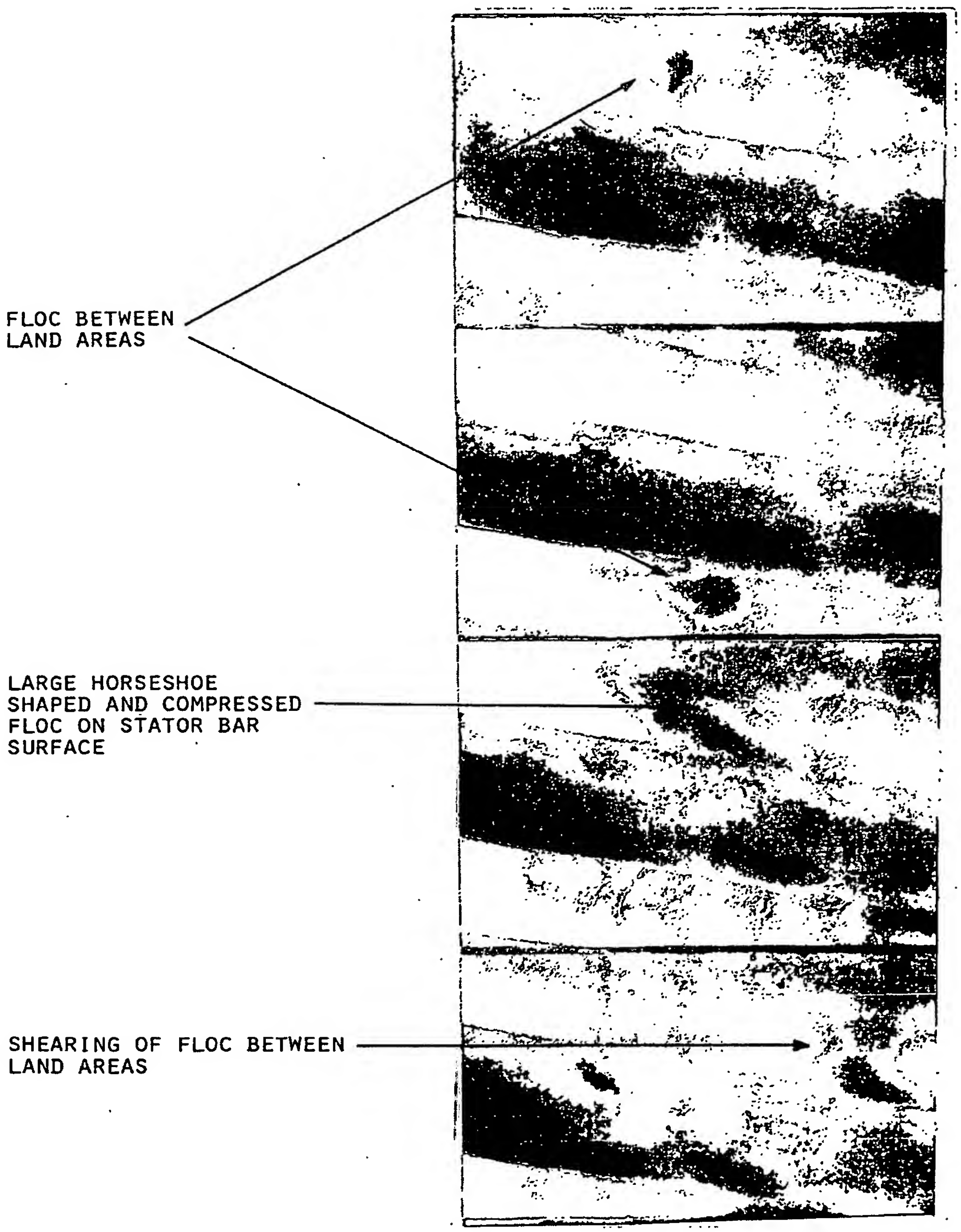


Figure 15. Enlarged 16 mm Film Frames Photographed Through Transparent Experimental Disc Refiner (Successive Frames Exposed at 1/1000 s)

the material suggests that fibers exist as a floc in between the bars. The floc covers only a very small fraction, perhaps around 10%, of the total area of the bar crossing. Thus one arrives at the conclusion that the pressure supported by the flocs could be up to 100 times higher than the average vectorial pressure between the plates. In other words, the flocs may be under a compressive stress of well over 10 MPa (100 atm.). This high stress can easily squeeze water out of the cell wall of the fibers even during the short period of a bar crossing ($\sim$0.2-0.5 ms). The layered structure of the cell wall will be exposed to enormous forces.

Based on the above analysis, it can be postulated that the compression induced movement of water inside the cell wall is a primary factor, contributing to the so called internal fibrillation. Similarly, it can be postulated that the estimated compressive forces on a cell wall level are high enough to cause cutting of fibers.

The size of flocs at the 1.1% consistency used may be estimated from the flocs visible in the stator grooves. The larger flocs seem to be around 4-6 mm long and 3-4 mm wide. It is not easy to get such a large "particle" to go into the narrow gap.

The flocs do not seem to stay on the stator land area for more than one passage of the rotor bar edge. It is impossible to conclude if the flocs adhere preferably to the leading rotor bar edge as postulated by Fox and coworkers (153,154)(Fig. 10).

This new direct evidence is in line with the hypothesis originally presented by Banks (119).

CONCLUSIONS

As a result of a critical review of the refining literature, one has to conclude that there seems to be a greater unity between the authors about the results of refining action on fibers, i.e., about the primary beating effects, than about the mechanisms which are actually contributing to the formation of the primary beating effects. In other words, the actual mechanics of refining, i.e., the understanding of how mechanical energy is transferred into the fibers and how this transfer causes the various structural changes in the fiber cell wall, is still very speculative. The main reason for speculation seems to be the difficulty of obtaining direct experimental results concerning the mechanics of refining.

Since energy is required for refining, the need for an adequate theory of refining is obvious: with a theory one might be able to increase the efficiency of refining considerably, and obtain combinations of refined fiber properties which are unattainable with today's refining techniques.

One reason for the lack of a theory of refining may be the underestimation of the network and flocculating character of the pulp to be refined. In most of the publications dealing with mechanisms of refining it seems to be assumed that fibers are treated individually and independently in the refining zone. If one accepts the idea that the fibers are flocculated, even possibly turbulent flow conditions existing inside the refiner, then it is easy to extend the fibrage theory of refining into the proposed hypothesis "Treatment of Flocs."

The flocs in refining may be visualized to have a dual role. First, the flocs, because of their large size in respect to the gap dimension, decrease the probability of treatment of fibers in the refining zone, i.e., the flocs decrease the efficiency of refining. Secondly, because a floc is an assembly of a very large

number of fibers, it protects many of the fibers from a catastrophic refining treatment, a result which is obtained if the refining is done at stock consistencies below 1%.

The reasoning leads to the following chain of thoughts. In order to increase the efficiency of conventional LC-refining, one should treat fibers instead of flocs inside the refiner. The refining zone should be designed so that all the fibers entering into it have a high probability of treatment. In order to be able to control the refining action specifically, one probably needs several types of refining zones; each of them being tailored to obtaining one or two specific primary beating effects. In order to increase the energy efficiency of today's LC-refining, the treatment zone should be decreased in volume considerably, so that the refining work to be done is concentrated to the small volume of the cell wall, thus minimizing the loss of energy to unnecessary turbulence and to unnecessary deformation of the fibers and flocs. It may also be necessary to develop new materials for the attrition surfaces, matching their properties to the elastic properties of the cell wall structure, which is to be modified by refining.

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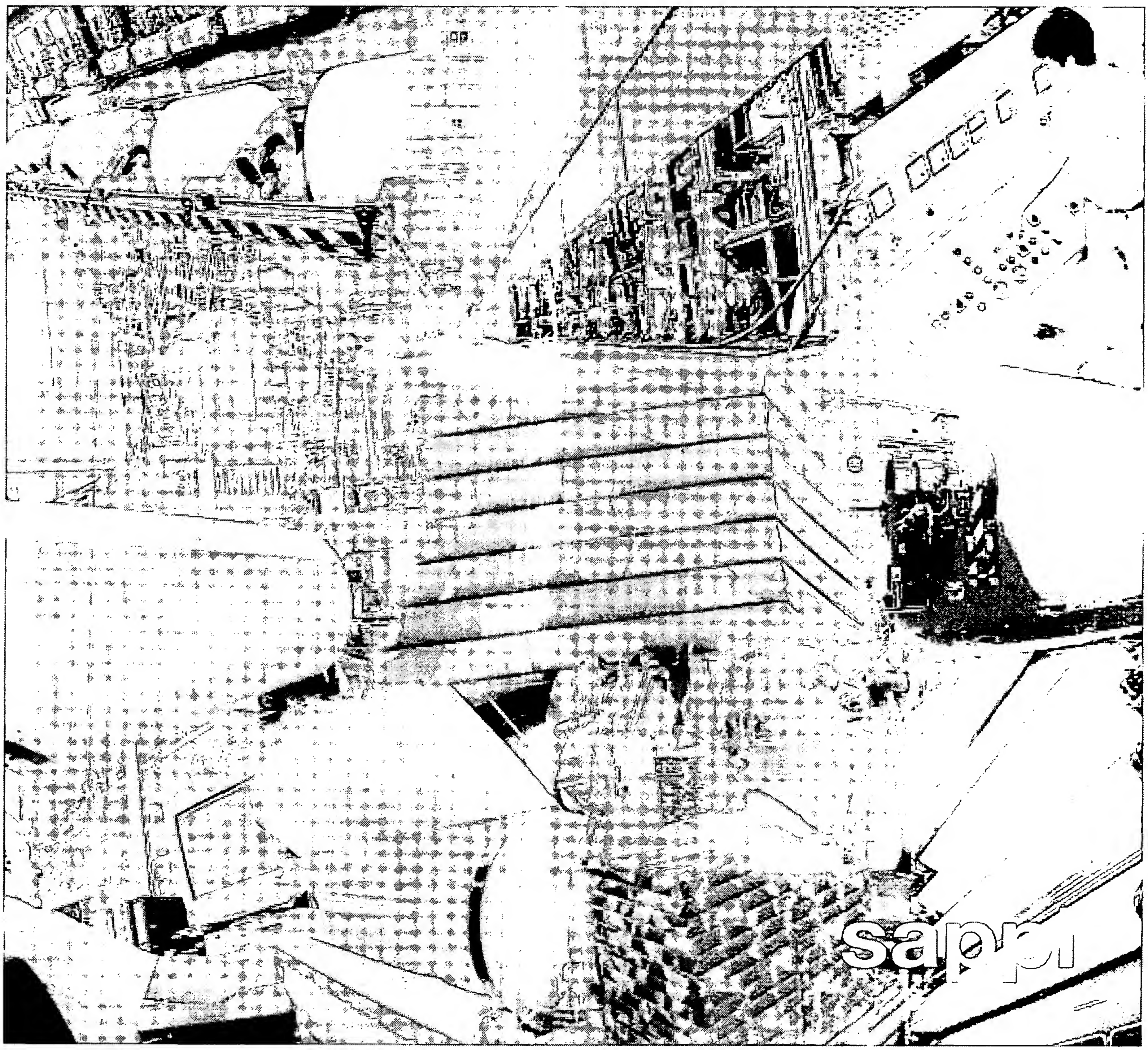
The Paper Making Process

From wood to coated paper



The Paper Making Process, the fifth technical brochure from Sappi Idea Exchange

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The Paper Making Process

From wood to coated paper

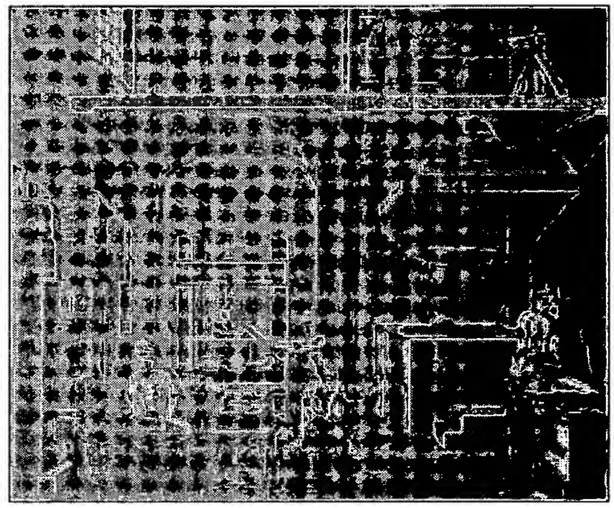
I Introduction

Though we may take it for granted, paper is always with us, documenting our world and reminding us of the limitless possibilities of life. Invented by the Chinese 2,000 years ago, paper has been used ever since as a communication medium. Initially, paper was made out of fibres from mulberry bark, papyrus, straw or cotton. Wood only emerged as the chief raw material for paper mass production as recently as the mid 19th century.

The printed page is immediate, its message cutting across cultures; a tactile experience that demands attention and creates desire. It is a passport to knowledge, a storage medium, a persuasive tool and an entertaining art form. Paper is a sustainable resource and a permanent document. It is the universal medium on which we chronicle our every-day history. Paper carries the past. It is the canvas on which we live the present and the blueprint upon which we design our future.

Paper touches the lives of every individual on this planet, and at Sappi, we never stop thinking about this fact. We are proud that Sappi is the largest and most successful producer of coated fine papers in the world. At Sappi, we are relentlessly developing new standards for the paper industry.

Drawing on centuries of experience, and the craftsmanship and expertise of its own people supported by 21st century papermaking technology, Sappi will lead the industry to ensure that this creative communication medium, paper, is the best it can be!



The interior of a historical paper mill

This brochure shows how we make this first class paper. Starting with the production of the most important raw material, wood. The pulping process converts this wood into the most appropriate type of pulp. The paper machine then converts the pulp into a thin base paper, which, at the end of the production process, is coated to give it a superb flat surface and bright shade. Following the description of this process, we will take a look at the properties used to measure the quality of paper.

II Wood production

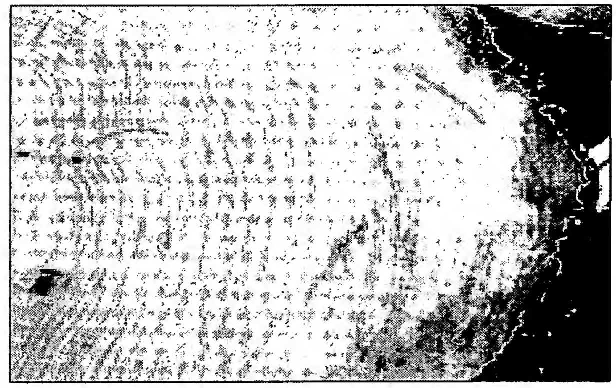
Wood as the raw material

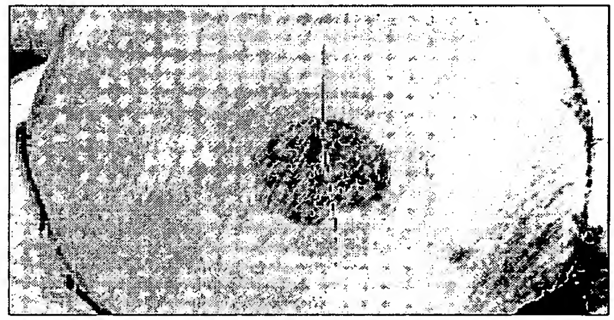
Approximately 25,000 plants with a woody stem are registered under the term wood. However, the different varieties clearly differ in terms of usability for the production of paper.

Conifers are preferred as the fibres are longer than, for example, fibres of deciduous trees. Longer fibres form a firmer fibrous web and, hence, a firmer paper on the paper machine.

Conifers used are mainly spruce, fir and pine, whereas beech, birch, poplar and eucalyptus are the most important deciduous varieties used for paper.

A trunk of a tree is not a homogeneous body composed of identical cells. The cells differ according to type, age, season of origination and arrangement in the trunk. At the outside, there is the bark, below are the bast and the cambium, which form the growth tissue. By cell division, the cambium grows out from the centre of the tree. Growth stagnation during the winter months results in the annual rings. The trunk with its different cells which are responsible for the transport of the nutrients and the saps can be used for paper pulp, but not the bark.

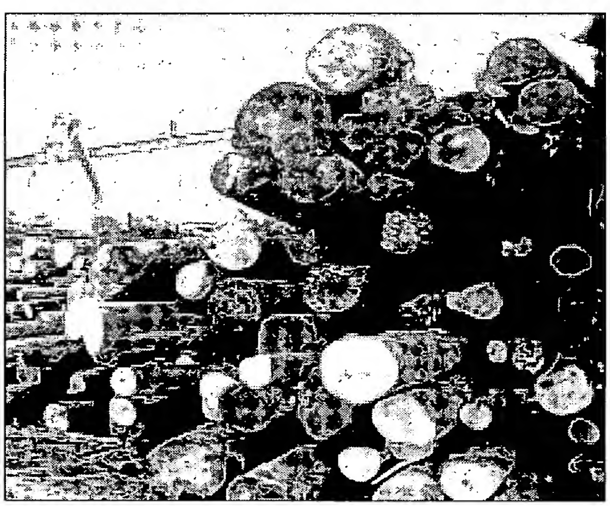




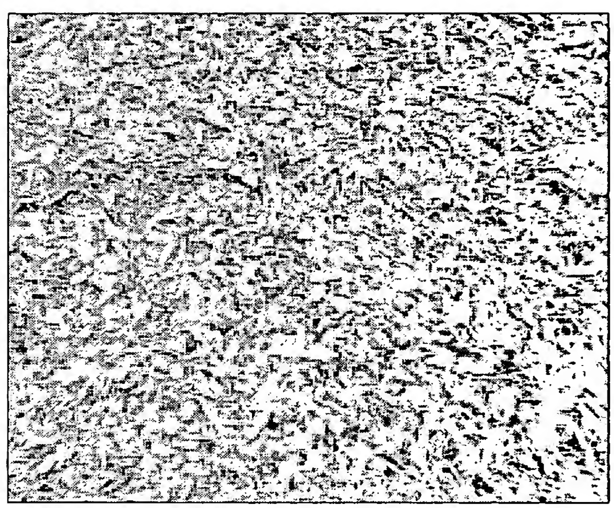
Beech trunks

This means that the wood supplied to the paper mill has to be debarked before it can be used to produce one of the varieties of pulp – the base material for the production of paper. The debarked trunks are either pulped to fibres (mechanical wood pulp) or processed to chips for chemical pulp.

The wood finds it way directly to the paper mill in the form of trunks or in the form of timber mill waste (slabs, chips).



Wood trunks



Chips

III Pulp production

Pulping process

Pulping of wood can be done in two ways: mechanically or chemically.

Mechanical pulp

In the case of mechanical pulp, the wood is processed into fibre form by grinding it against a quickly rotating stone under addition of water. The yield* of this pulp amounts to approx. 95%. The result is called wood pulp or MP – mechanical pulp.

The disadvantage of this type of pulp is that the fibre is strongly damaged and that there are all sorts of impurities in the pulp mass. Mechanical wood pulp yields a high opacity, but it is not very strong. It has a yellowish colour and low light resistance.

Chemical pulp

For the production of wood pulp, the pure fibre has to be set free, which means that the lignin has to be removed as well. To achieve this, the wood chips are cooked in a chemical solution.

In case of wood pulp obtained by means of chemical pulping, we differentiate between sulphate and sulphite pulp, depending on the chemicals used. The yield of chemical pulping amounts to approximately 50%. The fibres in the resulting pulp are very clean and undamaged. The wood pulp produced by this process is called woodfree. It is this type of pulp which is used for all Sappi fine papers.

The **sulphate process** is an alkaline process. It allows for the processing of strongly resinous wood types, but this requires expensive installations and intensive use of chemicals.

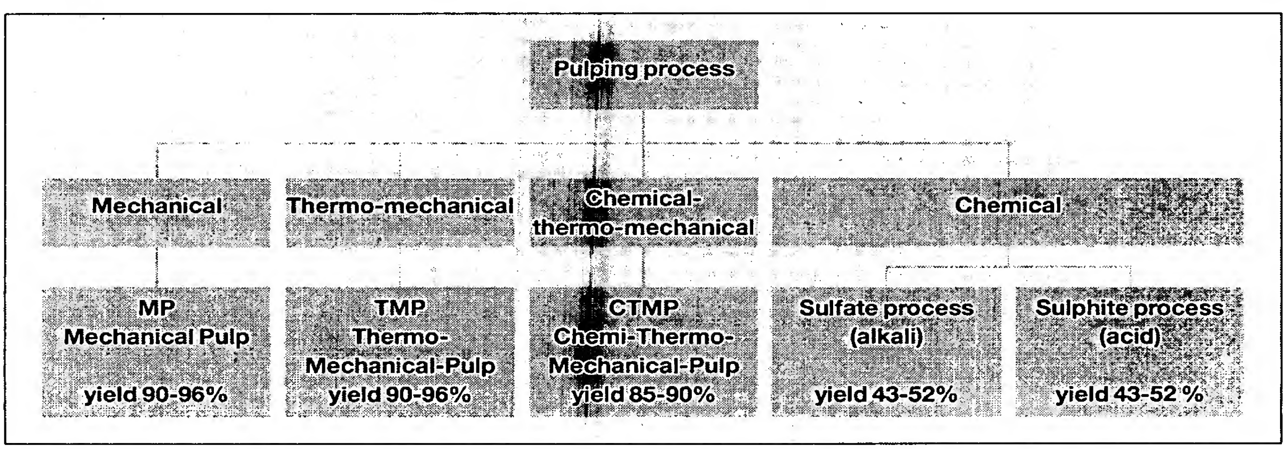
The **sulphite process** utilises a cooking acid consisting of a combination of free sulphur acid and sulphur acid bound as magnesium bi-sulphite (magnesium bi-sulphite process).

In the sulphite process, the cooking liquid penetrates the wood in the longitudinal direction of the fibres, which are aligned in this same longitudinal direction in the chips. When the cooking liquid penetrates the wood, it decomposes the lignin, which, during the actual cooking process, is converted into a water-soluble substance that can be washed out. The decomposition products of the carbohydrates are included in the cooking liquid as sugar.

When the waste fluids are concentrated in order to recycle the chemicals, these sugars are processed to alcohol and ethanoic acid. In this stage, the sulphite pulp is slightly brown and therefore has to be bleached to obtain a base colour suitable for white papers. This bleaching process, in which no chlorine or chlorine compounds are used, also takes place in the pulp mill as an integrated part of the overall operation.

The strength of sulphite pulps is less than that of sulphate pulps. Sappi uses only the magnesium bi-sulphite process in its own pulp mills.

** yield = usable part of the wood*



Intermediate pulp types:

TMP Thermo Mechanical Pulp
In this procedure, chopped waste wood is vaporised and then beaten into single fibres in refiners under vapour pressure.

CTMP Chemi-Thermo Mechanical Pulp
(wood pulp)
This process consists of a combination of impregnation (mixing with a chemical pulp), cooking, refining and bleaching. The pulping yield amounts to 90%.

The fibre length and the related strength of the paper are controllable. CTMP contains a certain amount of lignin, a tenacious, tough substance from the cell wall of the wood which strongly turns yellow.

Pulp bleaching

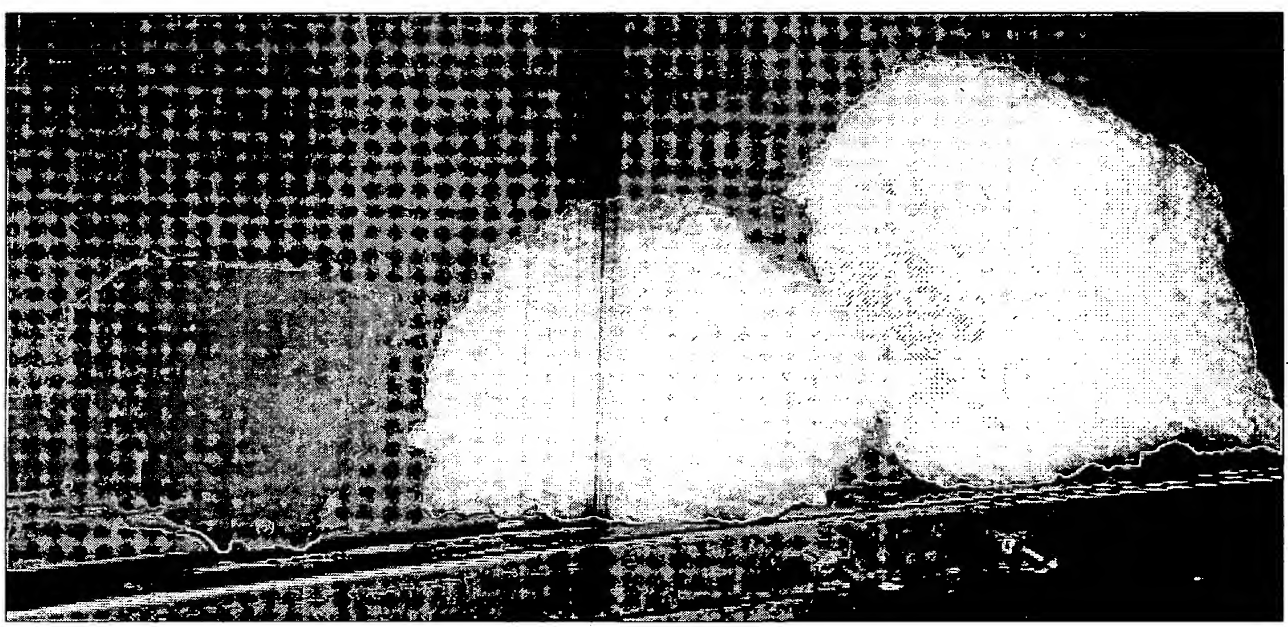
Initially, wood pulp has a brown or brownish colour. To obtain the brightness required for white papers, it has to be bleached. During this process of bleaching, the remaining lignin is removed as well. In practical terms, bleaching is a continuation of the chemical cooking process, taking place directly afterward in the pulp mill as an integrated next step of the overall procedure. Bleaching is a complex process, consisting of several chemical process steps, with washing taking place between the various chemical treatments.

The wood pulp can be bleached with chlorine / chlorine compounds, ozone / oxygen in different forms as well as hydrogen peroxide.

Based on the negative impact of some chlorine containing decomposition products, there are, however, environmental objections against the use of chlorine and chlorine products.

For this reason, Sappi has long ago switched to chlorine-free processes.

These processes are referred to as Totally Chlorine Free (TCF).



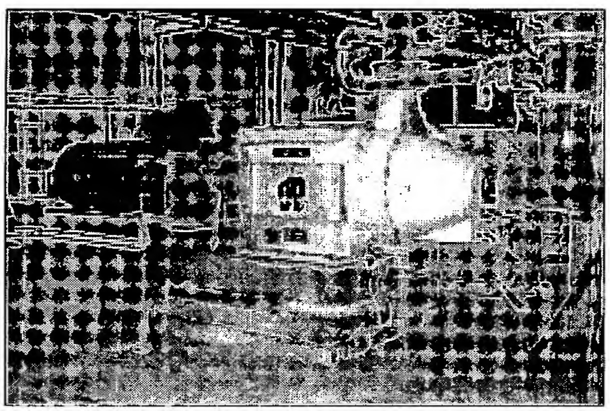
From the unbleached to the bleached pulp

IV Paper production

Raw materials

Preparation of the fibres in the refiner

The type of refining which takes place in the refiner has a decisive influence on the properties of the paper to be produced. A refiner is a refining aggregate with rotating and stationary cutters, the so-called rotors and stators. The variable positioning of these rotors and stators in relation to each other determines whether the fibres are being cut (free stock refining) or fibrillated (wet refining). Fibrillating is a fine bleeding of the fibre ends, resulting in a close-knit connection between the individual fibres. In the final paper this, in turn, results in greater strength.



Refiner for stock preparation

Additional raw materials for the base paper

Process materials include water, fillers, sizing substances, dyes and additives.

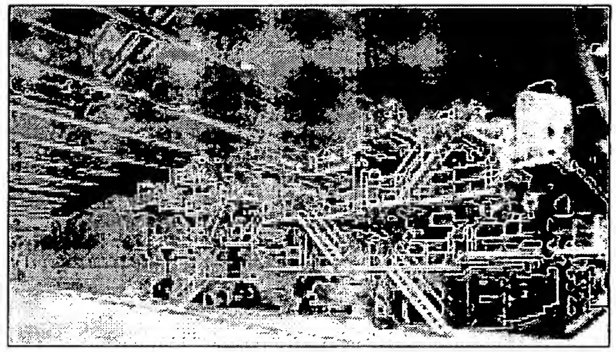
Fillers serve multiple purposes: they make the paper more opaque, more closed in its surface, brighter in shade as well as softer and more flexible depending on the requirement. Besides minerals, such as kaolin and china clay, the modern production process of paper makes extensive use of calcium carbonate (chalk), which has the additional advantage of making the paper more resistant to ageing. The total percentage of fillers used can be as high as 30% of the stock. In industrial paper production, the respective quantities and density ratios are regulated by computer controlled proportioning systems. This is the only way to guarantee a uniform quality standard in the production of high-quality brand papers.

But by far the most important process material is water. For each kilo of paper approximately 100 litres of water are required. In our time, the only justification – economically as well as ecologically – for the use of such enormous quantities of water, is closed circulation and effective waste water treatment.

The Sappi paper mills have the highest expenditures for environmental protection, even when compared to the high national standards.

In the proportioning system water, stuffs and fillers are brought together in mix tubs. The so-called constant part of a paper machine constitutes the transition from pulp preparation to the headbox of the paper machine. Another element of this constant part is the sorting unit, where impurities, foreign substances and patches are removed.

Fillers:	Calcium carbonate, Clay, Titanium dioxide
Additives:	Dye, Optical brightening agent
Binders:	Latex and starch products



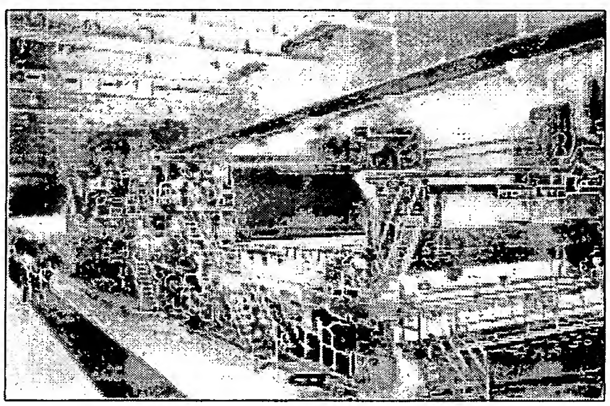
Gratkorn PM 11

Paper machine

Suspension at the headbox

After dilution and sorting in the constant part, the suspension of fibres, process materials and water has to be led to the wire part uniformly distributed across the width of the paper web. In principle, the speed at which the suspension exits from the headbox onto the wire has to be equal to the speed of the wire on which the sheet is formed. To achieve this, pressure is applied to the suspension in the headbox, in order to accelerate it to the wire speed. Apart from that, turbulence is generated just before the exit point of the headbox to avoid harmful flock formation.

The suspension leaves the headbox at the discharge lip. At this point, the suspension flowing onto the wire can have a thickness of up to 18 mm.



Ehingen PM 6

Sheet formation in the wire section

Once the suspension has left the headbox and comes into contact with the wire, the paper fibres move to the wire as a result of their natural flow resistance, thus forming a layer of fibres on the wire which accumulates towards the top of the stock. At the same time, water drains away at the bottom, and this combination of processes leads to two different forms of sheet formation, depending on the freedom of motion of the fibres in the suspension: through filtration and by means of thickening.

Filtration

In the case of filtration, a sharp transition is generated between the fibre layer building up on the wire and the suspension above. In this liquid phase, the pulp concentration is nearly constant and the fibres can easily move to each other in the corresponding ratio.

Thickening

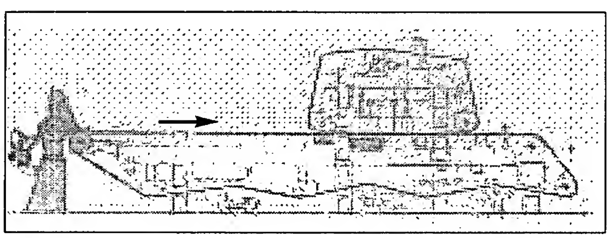
In the case of thickening, there is no clear division between the generated fibre mat and the suspension. The concentration increases linearly from top to bottom and the fibres are demobilised in the suspension. At the same time, water drains out from all layers of the suspension, to be collected for reuse.

The elements with which the sheet formation can be controlled are divided in four main groups:

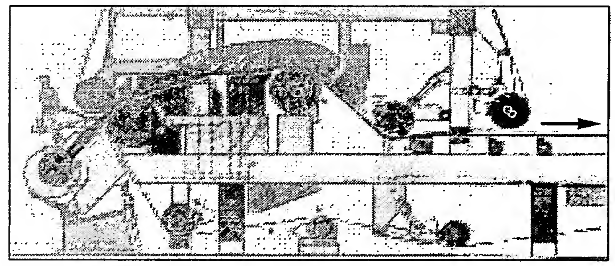
1. **Running elements**
   - ☐ the endless wire
   - ☐ the upper and lower wire
2. **Rotating elements**
   - ☐ table roll
   - ☐ forming roll
   - ☐ suction roll
   - ☐ squeeze roll
   - ☐ egoutteur
3. **Stationary elements**
   - ☐ wire table
   - ☐ hydro foil
   - ☐ vacu foil
   - ☐ suction box
4. **Mechanical elements**
   - ☐ screen adjustment of the headbox
   - ☐ wire shaking

Twinformer

Sheet formation takes place in the screen part of the paper machine. It is in fact an on-going battle between filtration and re-flocculation. The wire part can have different design features. The most common design is the endless wire concept. It is a universally applicable system allowing for high flexibility with regard to basic weight and sheet properties. However, these endless wire paper machines have a serious performance limitation in that they are strictly one-sided: drainage takes place only at the bottom, not at the top. And so, new designs were considered to increase drainage efficiency. This led to the development of the so-called twinformer, where additional equipment is installed on the rods of the endless wire. The twinformer is a design which provides for drainage of the suspension to the top side as well, by means of an added upper wire and a series of suction boxes. With this additional equipment, the paper stock can now be drained on both sides – from the bottom by means of gravity, and from the top, by means of suction. Drainage time is significantly reduced, which results in a far more efficient production process, with the added advantage of reduced two-sidedness of the paper.



Twinformer



Gapformer

Gapformer

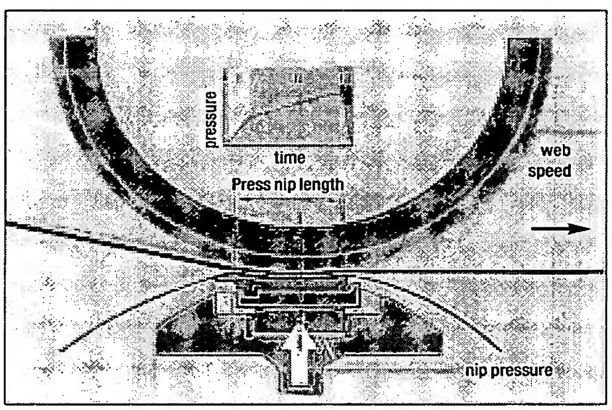
A further development in modern, high-speed paper machines are the so-called gap formers. In these formers, drainage is carried out to both sides simultaneously as the suspension is injected directly between the two wires directly from the headbox. Upon leaving the headbox, the pulp mass is immobilised in a matter of milliseconds, thus preventing later drainage elements from affecting the sheet structure which is now beginning to form. The fibre web is frozen – literally – the second it comes out of the headbox.

This process sets high demands on the quality of the headbox and the constant part.

De-watering in the press section

After formation of the sheet, a process which determines the most important sheet properties, the paper sheet has to be further drained and compressed. In this next phase, mechanical pressure exerted vertically to the sheet surface is used to further increase the proportion of dry content. In the press section, the web runs between a series of rolls which exert specifically set amounts of pressure. The water pressed out of the paper is absorbed by felts and transported off.

In recent years, shoe presses have been developed to increase the efficiency of the traditional roll presses. In these press units, one of the rolls is replaced by a hydraulically pressed shoe. This creates a bigger press nip, which makes the process more effective.



Shoe press

Dryer section

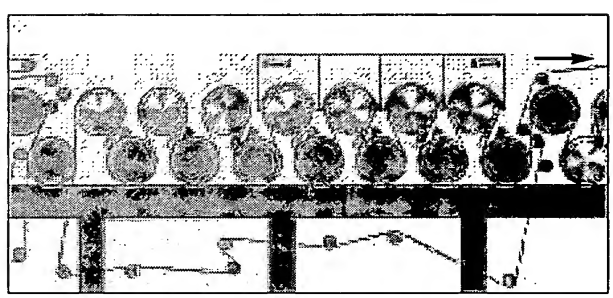
When the paper leaves the press section, it has a dry content of up to 50-55%. Now, the remaining water has to be removed by vaporisation. The most common type of paper drying is contact drying on cylinders heated with vapour. Here, the heat energy is transferred from the outside walls of the drying cylinders to the paper surface by direct contact. The dryer section consists of a succession of drying cylinders and the paper web is transported over and between these cylinders, the paper alternately making contact with the upper and the lower side. Drying takes place in different phases. In the short, first phase, only heat is transferred to the paper. There is no vaporisation. This takes place during the second phase, when the wet paper starts to convey its humidity to the surrounding air. In other words, the water contained in the paper starts to evaporate. In the third phase, the paper surface has already been dried to the maximum extent, and heat transmission into the dry paper stimulates vaporisation inside the paper.

End group

After conclusion of the drying process, the paper is often subjected to glazing in machine calenders. Besides machine calenders, which use steel rolls, there are also soft calenders, consisting of paired rolls where one is made of steel and the other one is coated with a soft, plastic material. This produces a better overall glazing effect and eliminates the problem of so-called "black glazing".

At the end of the machine, the paper is taken up on steel cores, the so-called tambours. Most paper machines use pope rollers. The tambour presses against the big pope roller and takes up the paper in uniform windings and at constant circumferential speed.

In the paper machine, there are various measuring frames at different positions, continuously measuring and controlling the selected quality parameters, such as base weight, moisture and ash contents, brightness and opacity.


Drying section

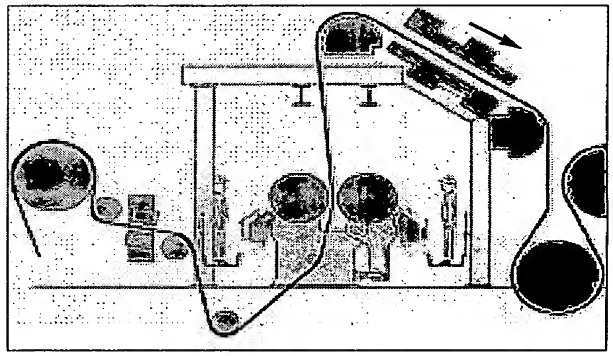
Surface treatment

Size press

The simplest form of refinement is surface treatment in the size press which is integrated in the paper machine.

Here, starch is applied to strengthen the paper surface. At the same time, this "closes" the surface of the paper, thus preventing problems like dusting or picking from occurring in the later printing process. In many cases, treatment in the size press is used to prepare the paper for the subsequent process of coating.

The size press consists of a pair of soft rolls, often coated with rubber, which press against each other as the paper web is guided through the nip between the rolls. The size solution is transferred to the paper through this nip, which also serves to control the dose of sizing being applied. In the size press, the quantity of applied pigments is limited.



Film press

Film press

As machine speed increased along with the quality demands for pigmented and machine coated papers, new multiple roll systems were developed which allowed for pre-dosing of the coating.

These modern press systems use a precisely pre-dosed film of coating, which is transferred to the paper in the nip between the application rolls. In this case, however, the coating is applied on the back side of the roll, using a design similar to that of a coating installation. These film presses run at high speed and they can operate with high concentrations in the sizing and pigmenting system.

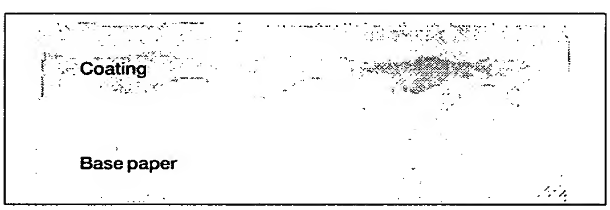
V Coating

The benefits of coated paper

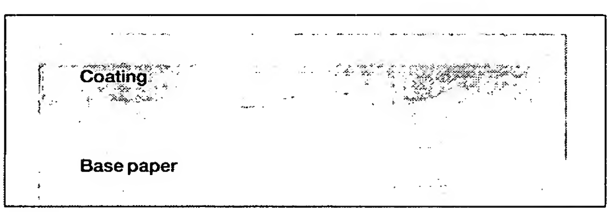
In recent decades, print media have had to meet increasingly high demands, in terms of visual aspects and in terms of printability. To meet these demands, coated papers were introduced many years ago.

Coating a paper enhances its optical and tactile characteristics – whiteness and shade, gloss and smoothness – but it also improves its printing behaviour, allowing the use of very fine screens, yielding more colour in thinner ink layers and producing more contrast in printed images.

When paper is coated, a covering layer of pigments, binding agents and process materials is applied to the surface. To achieve optimal results, all elements involved in the process must be perfectly tuned for mutual support, and this includes the coating colour, the coating method, the coating machine and its specific settings and the paper itself. One coating machine can apply multiple layers of coating, all depending on the intended use of the paper and all applications of coating requiring their own drying times. There are single coated papers, double coated papers and triple coated papers. In many cases, several methods of application are combined for an end result that benefits from each of the individual advantages.



Film coating



Blade coating

Coating machine

A primary reel, on which paper deficiencies can be removed, is superposed to the coating machine. To bridge set-up times at the coating machine, this primary reel has to operate at a higher speed than the paper machine. It has an unwinding system, designed for use of a flying splice. Next, the paper is coated. First on one side, followed by drying. Then the other side is coated and dried. For drying purposes, infrared-dryers, airfoils and drying cylinders are used. Rolls with gyratory grooves and wide plug-in reels provide safe reel guidance through the coating machine.

The heart of the coating machine, however, is the coating unit with the integrated coating aggregate. Under each coating unit, a workstation pumps up the coating mass from the preparation tanks, where the colour is mixed. These tanks, made of stainless steel, are cooled to avoid coating sticking to the walls and clot formation. The coating has to be permanently filtered and aerated to avoid deficiencies, such as blade streaks. Automatic control systems continuously monitor and adjust coating quantity and humidity of the coating.

At Sappi, different methods and techniques of coating are used. The two main coating techniques are film coating and blade coating.

In film coating and roll coating a uniform layer of coating is applied to the base paper. The surface contours of the paper remain visible. This is why the process is also known as "contour coating".

In blade coating, an excess layer of coating is first applied to the paper, which is then partially scraped off again ("doctored") with a steel blade. The pressure exerted by this doctor blade produces a uniform surface. The cavities of the paper are filled with coating and the fibre backs remain nearly uncovered.

Coating preparation

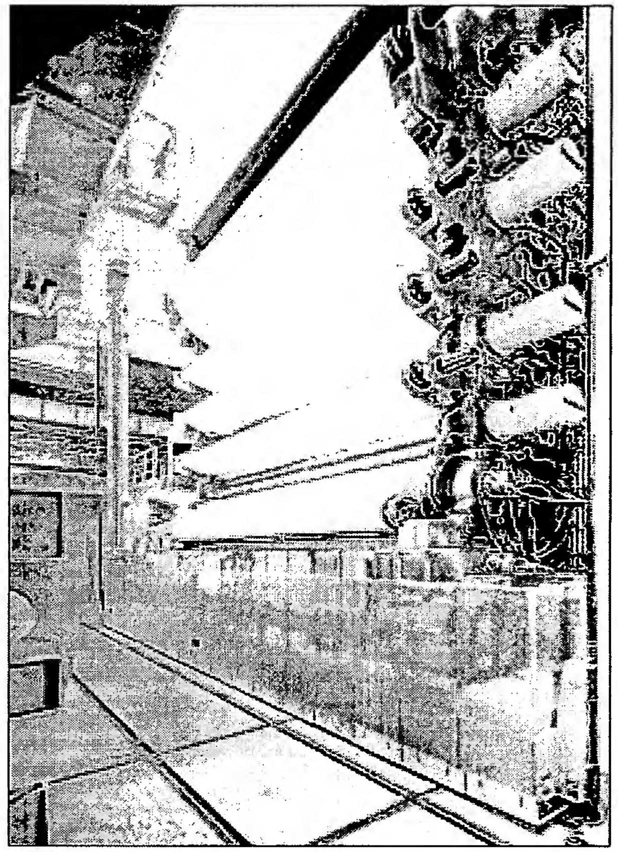
Coatings mainly consist of pigments (chalk, clay or talcum). In themselves, however, these are powdery substances which would be blown off the paper surface like dust. Therefore, binding agents must be used to provide adherence to each other and to the paper. Depending on the intended use of the paper and the type and structure of the pigments used, different quantities of binding agents are required. Binding agents can have a natural basis (casein or starch) or a synthetic composition (synthetic dispersions).

Process materials add specific properties to the coating. One commonly used process material is optical whitener. It converts invisible, ultraviolet light into visible bluish white light, giving an impression of true whiteness.

In the so-called "coating kitchen", these individual components, taken form large storage silos, are mixed in stainless steel tanks. After having been thoroughly screened, naturally. Each specific coating has its own recipe, exactly prescribing the quantities of each component. To preserve consistency from preparation to preparation, the whole process of coating production is fully automated.

In the coating units, measuring frames monitor the weight of the coating being applied to the paper and the resulting gloss of the paper itself.

Usually, the coating kitchen is also responsible for preparation of the starch solution used in the size press (with or without pigmentation), which is an integrated part of the paper machine.



Supercalender

VI Finishing

Calender

Calenders are used to make the paper surface extra smooth and glossy. A calender consists of a number of rolls, where pressure and heat is applied to the passing paper. There are many different types of calenders. Some can be integrated as part of the paper machine, others are operated separately, as stand-alone installations. These separate installations, the so-called supercalenders, can include up to 16 rolls. These rolls can have different surfaces – they can have steel walls or coverings with elastic materials, depending on the desired extent of glazing.

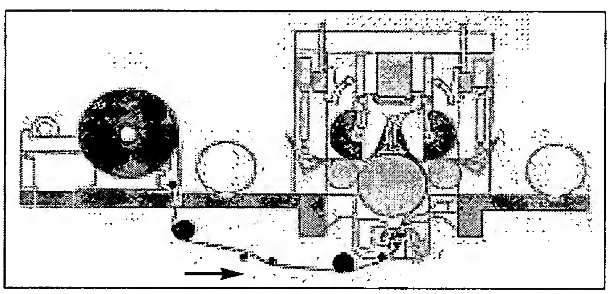
Depending on type, the paper is ready after it leaves the coating machine or the calender.

Rewinder

The function of the rewinder is to rewind the reels from one tambour to another tambour. Here, the web run can be changed, from the outer to the inner side, the reel edges may be cut and deficiencies in the paper can be removed.

Slitter rewinder

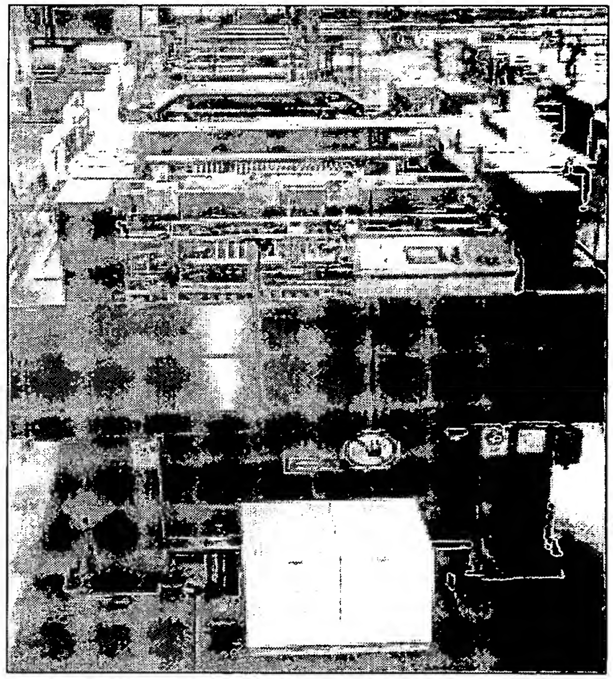
The finished paper, which on the tambour still has the full machine width, is cut to smaller reels on the slitter rewinder. Circular knives cut the tambour to reels of specified width while the tambour is being unwound. Depending on paper type, these reels are now ready for delivery to the customer, or they are transported to a cross cutter where the paper is cut to sheets.



Reel cutter

Cross cutter

In a cross cutter, the smaller reels that have been cut to size from tambours by the slitter rewinder, are cut to sheets of a specified size. Several reels can be processed simultaneously, depending on the design of the cross cutter and the "cutting weight" of the paper. The important thing here is to produce sheets with clean cutting edges, in other words, to prevent cutting dust from clinging to the edges, since this would cause problems in the printing process. The paper reels fed into the cross cutter are trimmed on both edges and separated in longitudinal direction by circular knives. The web is then cut off to the required size by the chopper knife.



Cross cutter

One important aspect is that the cutting process must be perfectly synchronised to produce the exactly right size and squareness. A conveyor belt directly after the knife section holds the sheets in position and transports them at high speed to a second conveyor belt. Here, the speed is reduced and the sheets are laid out in overlapping arrangement for further transport to the final stacked layout.

Modern cross cutters do more than just cutting. They check the quality of the paper surface, remove faulty sheets, count the sheets and insert counting strips. Some even allow for a "flying change" process of continuous operation, in which full pallets are automatically transported off and new pallets moved into position without halting the machine.

Guillotine

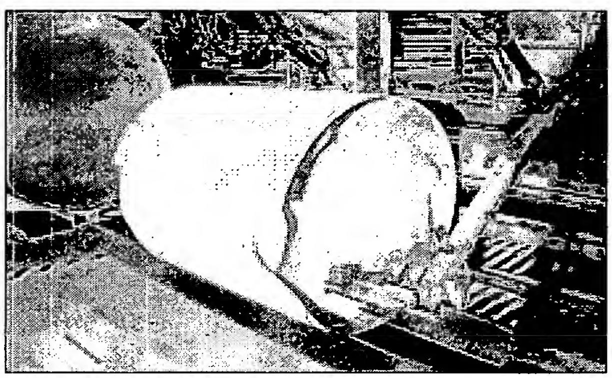
Guillotine type cutters are used for cutting relatively small quantities of paper in special sizes. In these cases, completely refitting the cross cutter would not be economically sensible. Guillotines are also used for the so-called four-sided trim which is necessary for certain print jobs.

VII Packing and storage

Finally, the paper is packaged for transport to the customer. The packing is important to avoid transport damages and to provide protection against moisture. Transport methods and means determine the type of packing.

Automatic reel packing machines perform the following tasks:

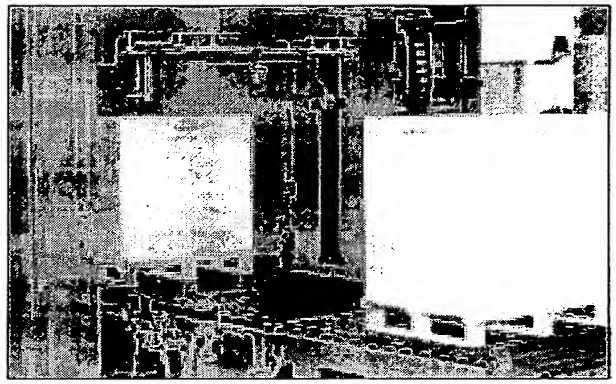
A bar code identifies the reel. While the reel is positioned in the centre, interior side caps are applied on both sides. Based on the sequence belonging to the bar code, a machine wraps the reel with pre-selected packing paper, using a certain number of windings and a specific type of gluing. Finally exterior side caps are added, the reel is weighed and the labels are attached. The reel then goes to the transport department via a conveyor belt.



Packing of reels

Sheetsize paper can be packed in reams or delivered as bulk-packed goods, with pallet packing only. Reams can contain 100, 250 or 500 sheets. In the case of smaller orders or special sizes, ream wrapping is carried out manually. Large stock orders in standard sizes are reamed on ream wrapping machines.

The packing material is designed according to customers' requests. Protection against damage and moisture is the main concern, but ream wrapping can be used as an advertising medium as well.



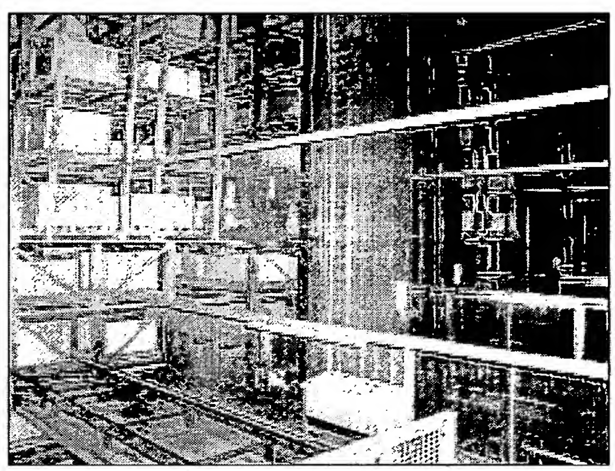
Packing of pallets

The defined packing units are taken off the stack before reaching the ream wrapping machine and transported in correct alignment to the first wrapping unit.

The packing paper is cut off, wrapped around the paper and glued in place. The packed reams are then stacked and labelled.

Pallets with sheetsize paper (reamed or bulk-packed) are wrapped vapour tight with shrinking or wrapping foil. For transport over longer distances, a covering plate is applied and the packed pallets are reinforced with loops of steel or plastic banding.

Pallets packed in vapour tight wrapping do not require a full air-conditioning when in storage. They can be stored in light and water protected areas. Paper producers and wholesalers often use high shelf stocks, in which the pallets can be stored randomly, to be picked by computer-controlled picking systems.



Automatic warehouse

VIII Paper properties

The data sheets list the most important quality characteristics of the paper.
These include:

Basis weight

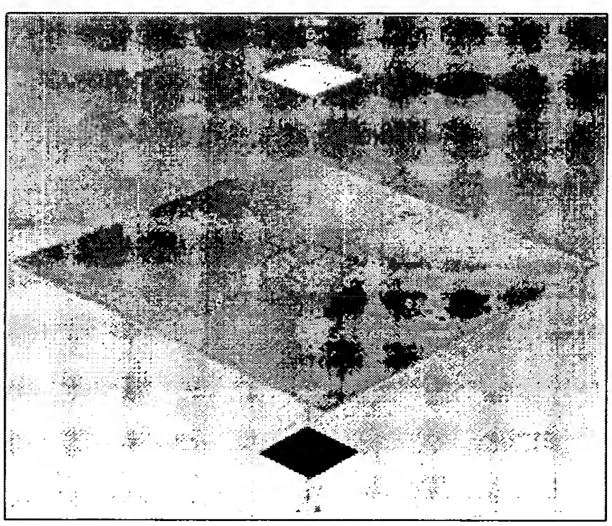
The basis weight of a paper means the weight in grams per square meter (g/m²) under conditioned circumstances. The entire mass is the sum of fibrous materials, fillers, process materials and water.

Brightness

The brightness (ISO) is a measure for the brightness degree of the paper expressed in percent compared with the brightness standard (magnesium oxide = 100%). The higher the brightness value, the brighter the paper is.

Gloss

The gloss figure in the data sheets indicates the percentage of reflected light with a defined angle of incidence. A higher gloss leads to stronger light reflections and higher gloss values.



Colour space

PPS roughness

The geometric form of a paper surface is defined as deviation from the ideal flat level. The more the surface approaches the ideal level, the smoother the paper is. The measuring method (PPS) is based on the measurement of air leakage between the paper surface and the even measuring head. In the case of PPS roughness, the average pore depth over a defined circular area is measured. The higher the measured value is, the "rougher" the paper surface is.

Opacity

The opacity is a measure for the opacity degree of the paper, expressed in percent in relation to the reflected light. Paper which lets a lot of light through, is transparent; paper that lets little light through, is opaque. The higher the value, the more opaque the paper is.

Relative humidity

At a given temperature, there is a maximum to the amount of water vapour that the air can absorb. Relative humidity indicates the percentage of this maximum which is actually in the air (i.e. between the sheets of a stack or the windings of a reel).

pH value

The value in the data sheets defines the pH value of the surface. The pH values are indicated on a scale from 0 to 14. The value 7 marks the neutral point which corresponds to distilled water. Values below 7 refer to "increasingly acid ", values above 7 stand for "increasingly alkaline". Papers should have a pH close to the neutral point in order to meet ideal requirements for printing and further treatment.

Specific volume

Paper thickness is expressed in micrometer (µm). To compare the thickness of papers with different basis weights, specific volume is used.

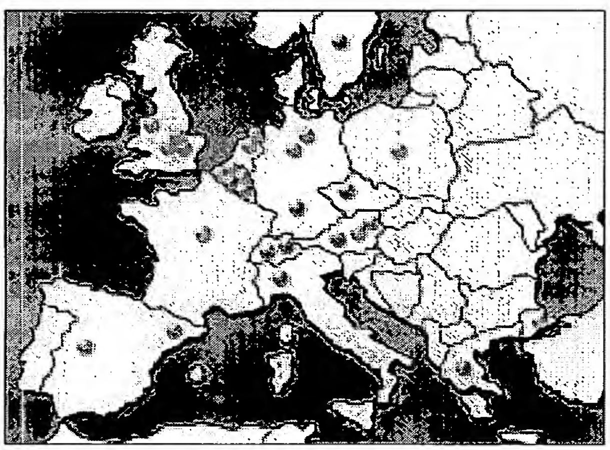
$$\text{volume} = \frac{\text{thickness (µm)}}{\text{basis weight (g/m}^2\text{)}}$$

IX Concluding remarks

The content of this brochure is based on our technical experience in papermaking and textual building blocks from the book "Het Papierboek".

We want to express our appreciation for this to:

EPN Publishers
Houten, The Netherlands



- **Sappi Fine Paper Europe head office**
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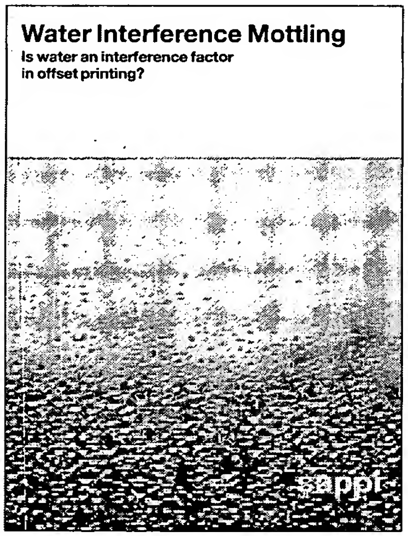
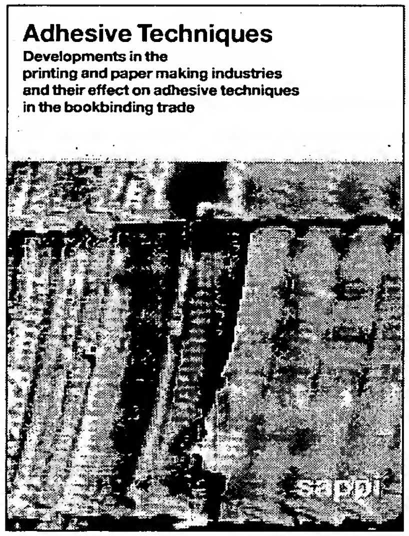
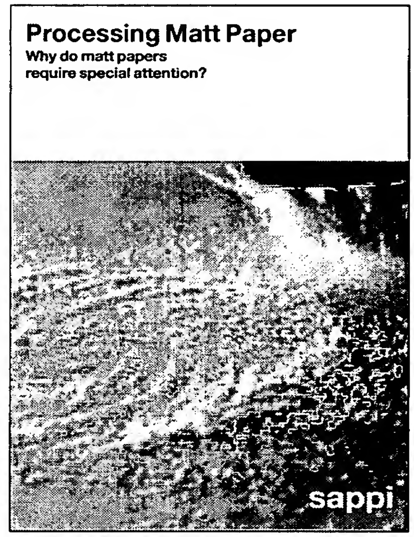
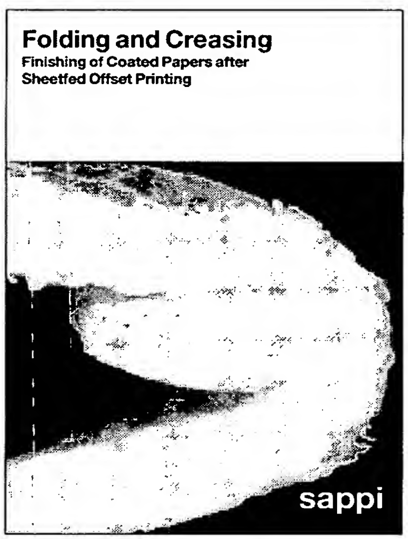
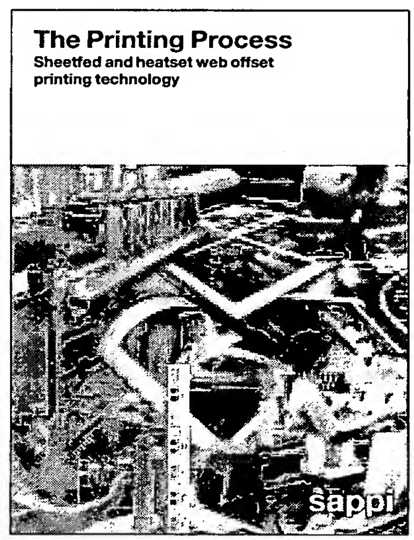
The Paper Making Process is one of Sappi's technical brochures. Sappi brought together this paper related knowledge to inspire our customers to be the best they can be.











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The Paper Making Process video and the other technical brochures are freely available at our knowledge bank:

www.ideaexchange.sappi.com/knowledgebank

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The word for fine paper

US005385640A

United States Patent [19]

Weibel et al.

[11] Patent Number: 5,385,640

[45] Date of Patent: Jan. 31, 1995

[54] PROCESS FOR MAKING MICRODENOMINATED CELLULOSE

[75] Inventors: Michael K. Weibel, West Redding; Richard S. Paul, Redding, both of Conn.

[73] Assignee: Microcell, Inc., West Redding, Conn.

[21] Appl. No.: 89,683

[22] Filed: Jul. 9, 1993

[51] Int. Cl.⁶ D21B 1/10

[52] U.S. Cl. 162/23; 241/21; 241/28

[58] Field of Search 162/23, 100; 241/21, 241/28

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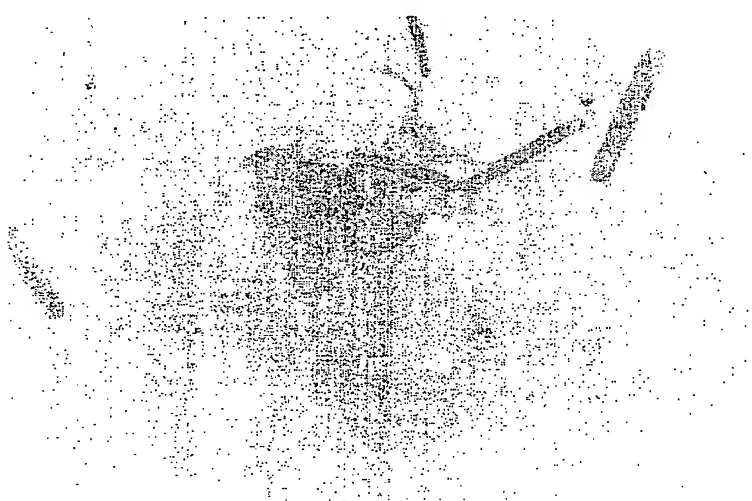
Primary Examiner—W. Gary Jones
Assistant Examiner—Dean T. Nguyen
Attorney, Agent, or Firm—Dann, Dorfman, Herrell and Skillman

[57] ABSTRACT

A process for the production of mechanically disassembled cellulose and the resultant product, referred to as microdenominated cellulose (MDC). The product is characterized by a settled volume of greater than 50%, as determined on the basis of a 1% by weight suspension in water after twenty-four hours, and a water retention value of over 350%. MDC is useful as an ingredient in foods, pharmaceutical and cosmetic products.

7 Claims, 6 Drawing Sheets



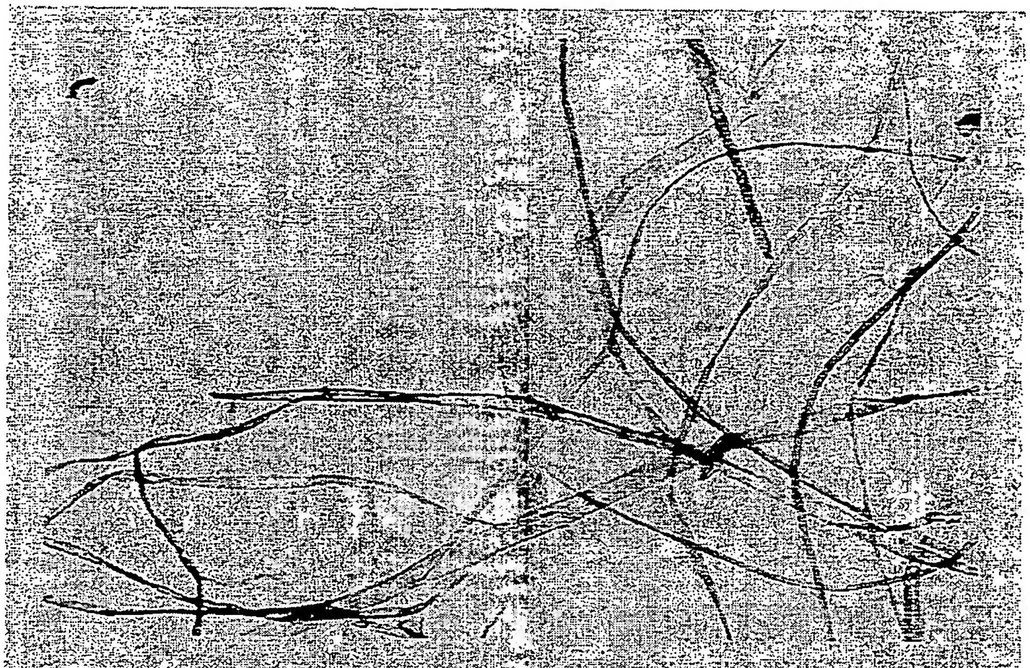


FIG. 1

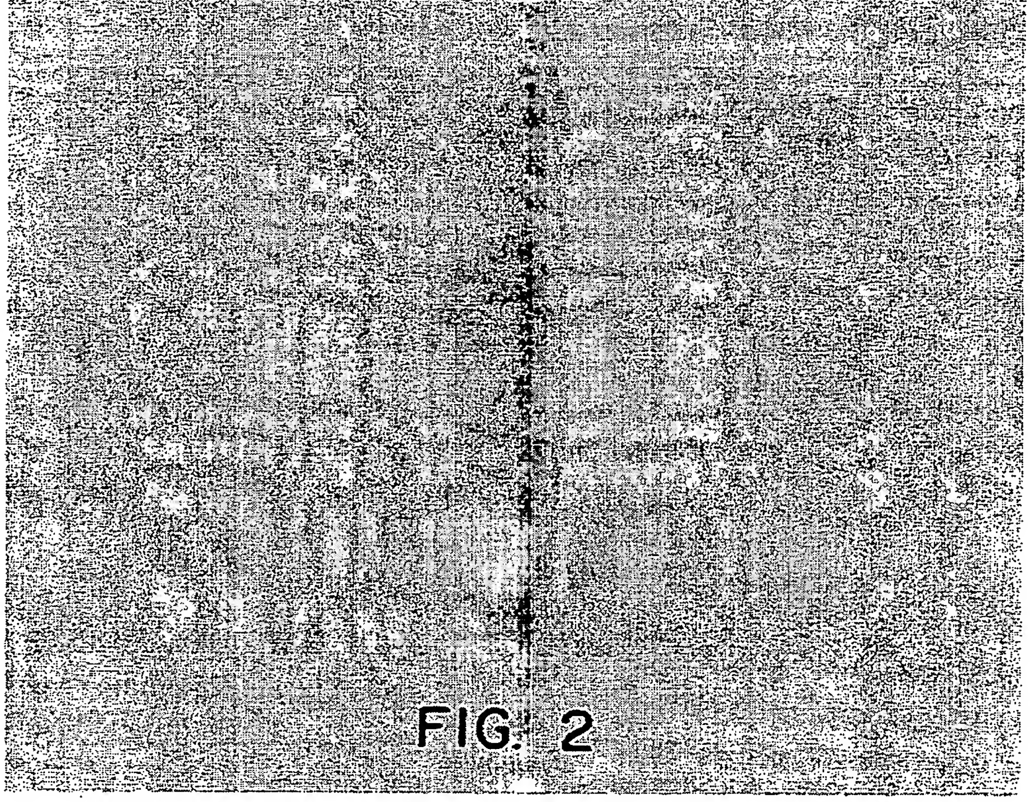


FIG. 2

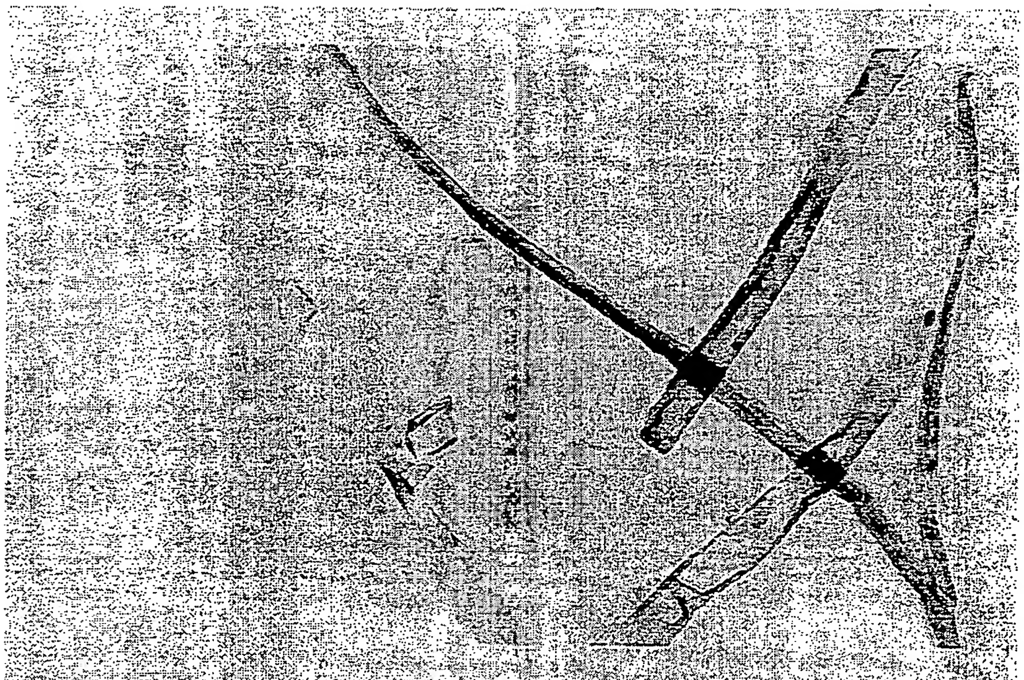


FIG. 3

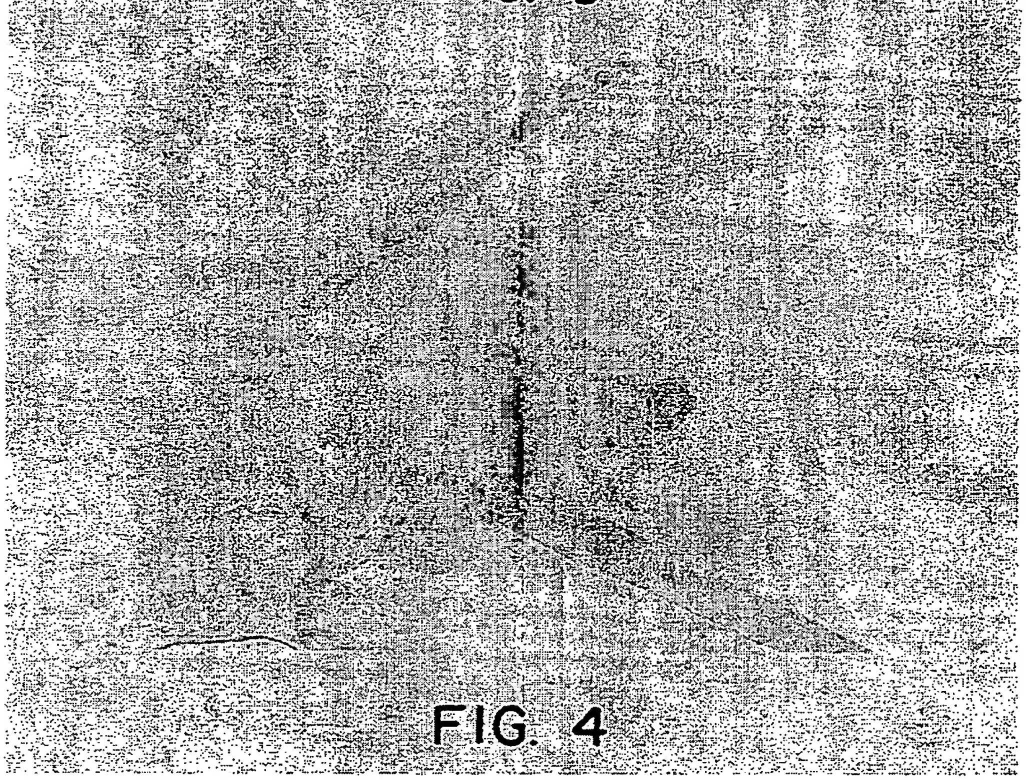


FIG. 4

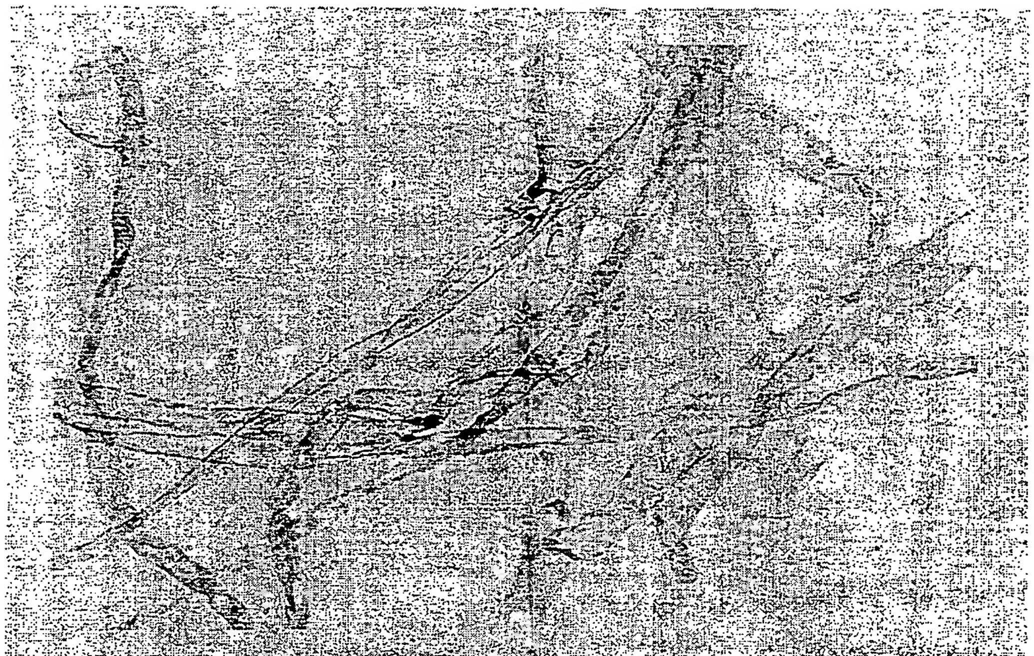


FIG. 5

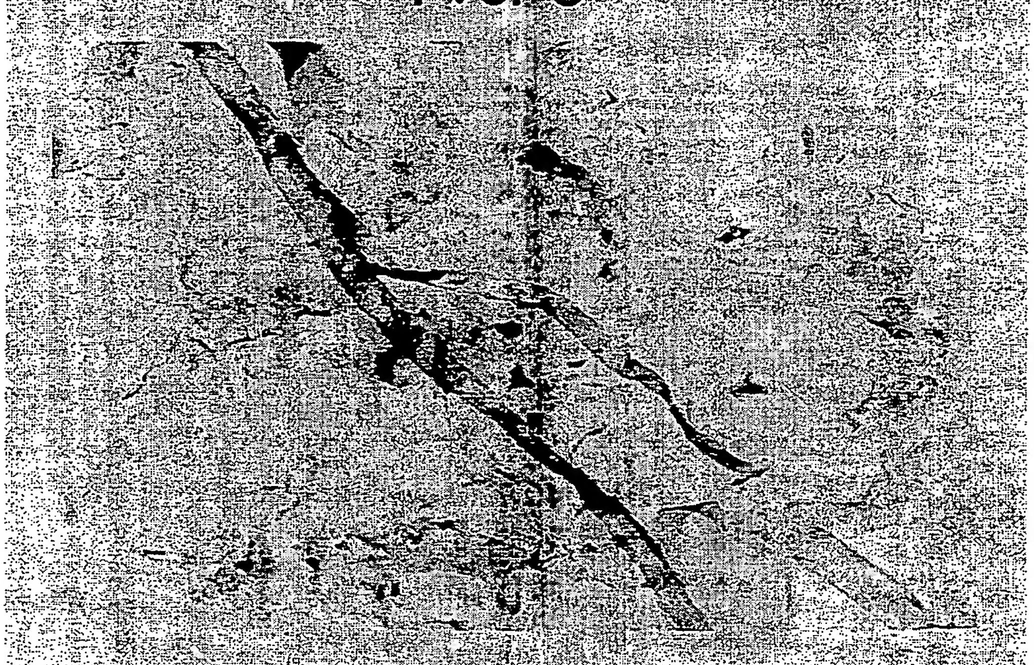


FIG. 6

FIG. 7

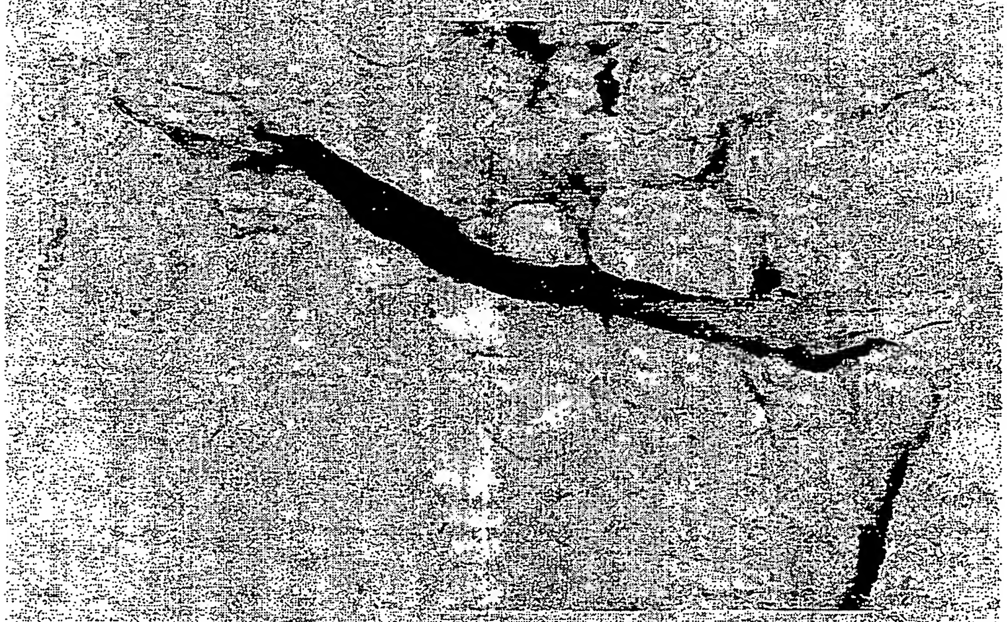


FIG. 8

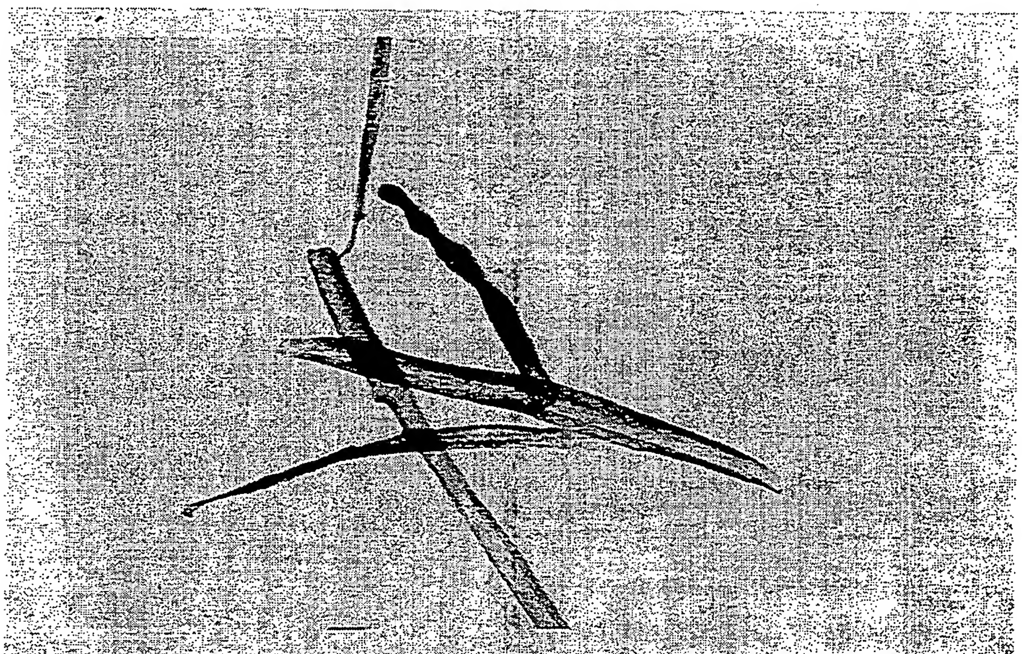


FIG. 9

FIG. 10

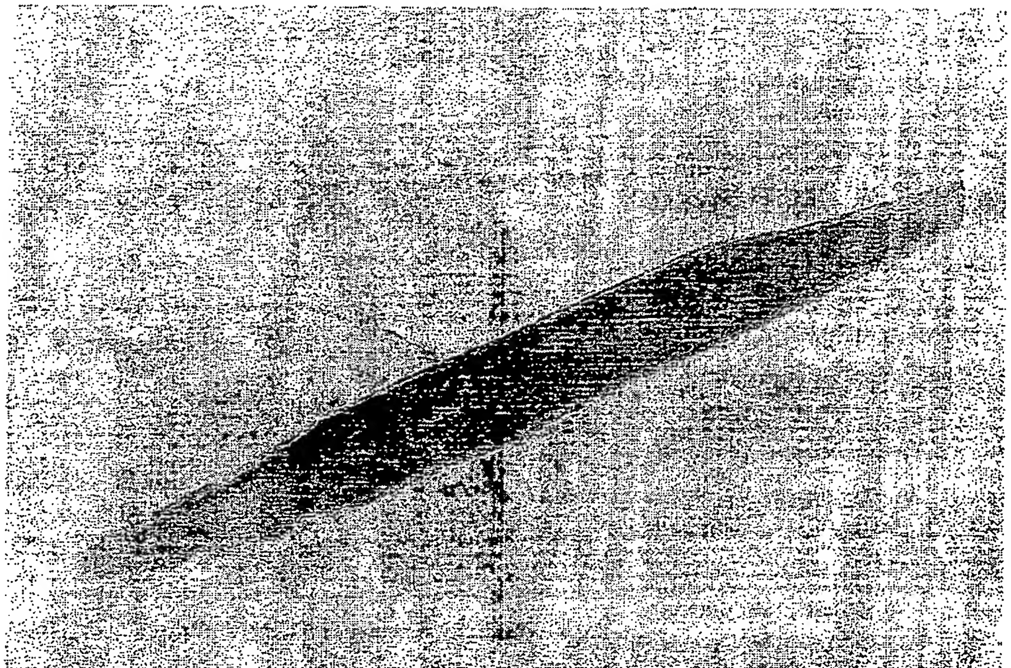


FIG. 11

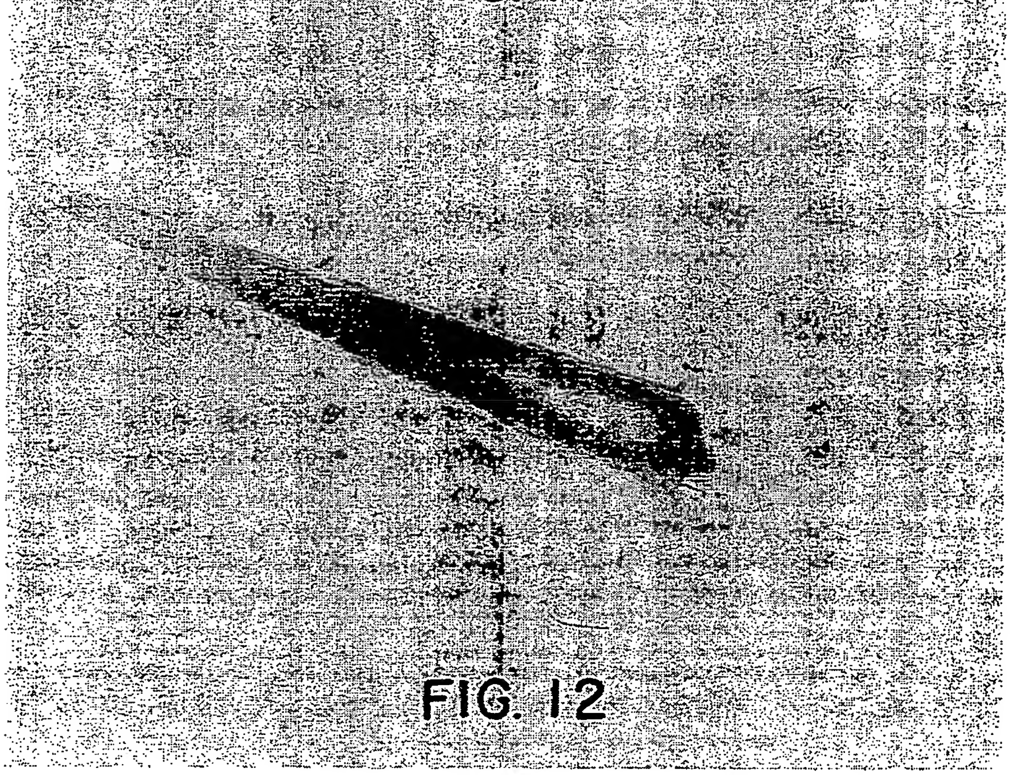


FIG. 12

PROCESS FOR MAKING MICRODENOMINATED CELLULOSE

This invention relates to microdenominated cellulose and to a process for its preparation.

BACKGROUND OF THE INVENTION

Since cellulose is the major structural constituent of most plant matter, it is natural that those interested in processing or refining such materials refer to them as cellulosics. However this general term connotes a multiplicity of meanings whereby each is qualified by descriptors frequently specific to the interest at hand. The commercial applications of processed plant matter to produce a refined cellulosic material are numerous and involve use in many nonanalogous arts. For example, refined celluloses are extensively used in paper and textile applications. Refined cellulose is also used in adhesives, food ingredients, industrial coatings and various other diverse applications. For each end use, the raw material, processing and final product(s) comprise a technological field essentially unique to itself.

In general, a wide variety of chemical, thermal and mechanical transformations are known in the art to refine, manipulate and modify cellulose for numerous purposes. The following hierarchical characterization has been devised to describe previously known technology relating to structural manipulation of refined cellulosic substances. This characterization serves the additional purpose of providing bases for distinction between the process of the present invention and the prior art.

The molecular level or primary structure of cellulose is the beta 1–4 glucan chain. All celluloses share this level of structure and it is the distinguishing difference between cellulose and other complex polysaccharides. The natural chain length is not known due to unavoidable modification and degradation which occurs during the disassembly to this level, but probably extends into the polymerization regimes of many thousands of glucan units. Transformations at this level of structure involve forming and breaking chemical bonds.

Secondary structure is considered to be submicroscopic strands formed from parallel, aligned assemblages of glucan chains. This level of organization is designated the microfibril. Microfibrils are spontaneously formed from a plurality of nascent glucan chains believed to be synthesized simultaneously by a complex, motile, biosynthetic organelle involved in the assembly of the primary plant cell wall. The microfibril is of sufficient size to be discernable with the electron microscope and depending on the plant species ranges in its major cross-sectional dimension from approximately 50 to 100 Angstroms. As with the beta-glucan chain, of which it is composed, the length is indeterminant. Noncovalent interactions, such as by hydrogen bonding, stabilize secondary structure. Because the interchain attraction is high, structural transformation is probably rare unless preceded by chemical modification of primary structure.

Tertiary structure is related to arrays and associations of microfibrils into sheets and larger stranded structures designated fibrils. The distinguishing features at this level of structure are sufficiently small that resolution is usually possible only via the electron microscope. However, some individual fibril assemblies are of sufficient gross cross-sectional dimension (0.1 to 0.5 microns or 10,000 to 50,000 Angstroms) to be discernible with the light microscope. Structural deformation at this level is largely mechanical and either organized (disassembly/denomination) or random (indiscriminate fracture/cleavage).

Lastly, quaternary structure deals with the construct of tertiary elements which form the primary and secondary cell wall. This level of structure defines the physical dimensions of the individual cell and any gross structural specialization required for physiological function of the differentiated cell. Examples are libriform, tracheid and parenchymal cell structure. Structural manipulation results from indiscriminent comminution and is the most commonly employed mechanical transformation practiced.

Conventional pulping of cellulosic materials is primarily concerned with chemi-thermomechanical processing of schlerenchymous or structural plant tissue to achieve individually dispersed cells. The result is a quaternary structure largely consisting of cellulose derived from the primary and secondary cell walls. Depending on the plant source and extent of processing some heteropolysaccharides such as hemicellulose (xylans, galactomannans, pectins, etc.) may also be present. The important distinction of pulping from other processing of celluloses is that an anatomical destruction of intact plant tissue occurs. This results in dispersed cellforms which represent a minimal degree of quaternary and more basic structural levels of manipulation. Some forms of cellulose, such as cotton, are produced naturally in a dispersed state and do not require pulping as a prerequisite.

Important to the following discussion is the distinction between disassembly and indiscriminate fragmentation processes. In fragmentation the localized energy excursion (by whatever means) is sufficiently high and accumulates sufficiently rapidly that an organized dissipation of internal energy by the acquiring matrix is not effected. Here an intense perturbation is applied and results in an indiscriminate fracture or other major disorganization at translocations within a defined microdomain. In the case of disassembly, on the other hand, the acquired energy excursion is dissipated in a more organized manner usually following a path of lowest activation energy. For cellulose this appears to involve segmentation along parallel fibril oriented assemblies and possibly laminar sheet separation of fibril arrays.

Mechanically beaten celluloses have long been employed in the paper and packaging industry. Chemithermomechanically refined wood pulps are typically dispersed in hydrobeaters and then subjected to wet refining in high speed disc mills. This level of structural manipulation as presently practiced is exclusively at the quaternary level. The objective of such processing is to disperse aggregated fiber bundles and increase available surface area for contact during drying to increase dry strength. Substantial size reduction and concomitant impairment of dewatering are undesirable and circumscribe the extent of processing. The measurement of the ease of water drainage from a beaten pulp is termed Canadian Standard Freeness and reflects the ease or rate of interstitial water removal from the paper stock.

Finely ground or fragmented celluloses are well known. These products are produced by mechanical comminution or grinding of dried, refined cellulose. They are employed largely as inert, non-mineral fillers in processed foods and plastics. The manipulation is

exclusively at the quaternary level of structure. It is achieved by application of a variety of size reduction technologies, such as ball and bar mills, high speed cutters, disc mills or other techniques described in part in U.S. Pat. No. 5,026,569. The practical limit of dry grinding is restricted in part by the thermal consequences of such processing on cellulose and in part to the economics of equipment wear and material contamination of the product. Micromilled cellulose (MMC) prepared in aqueous or other liquid media as described in U.S. Pat. No. 4,761,203 avoids the thermal decomposition associated with prolonged or intense dry grinding. This technique allows particle size reduction into the colloidal range (about 10 microns). It is believed to operate by indiscriminate micro-fragmentation of quaternary structure, without incurring the fusion/thermal degrading effects characteristic of dry grinding.

Microfibrillated cellulose (MFC), as disclosed by Turbak et al (U.S. Pat. No. 4,374,702), is principally a mechanical manipulation of refined cellulose from wood pulp at the tertiary level of structure. The process employs high pressure, impact discharge onto a solid surface of a cellulosic dispersion in a liquid medium. This results in a combination of direct energy transfer through high, adiabatic shear gradients generated within the impact domain and secondary effects of such shear (or translational momentum exchange) from solvent cavitation to disassemble suspended cellulose particles. Depending on the extent of processing and preconditioning of the raw material the structural manipulation produces fibril ensembles of disassembled quaternary structure. These highly dispersed fibril structures impart unusual properties to the continuous liquid phase in which they are prepared.

Microcrystalline cellulose (MCC), as disclosed in U.S. Pat. No. 3,023,104, exemplifies structural manipulation which can occur at the secondary level of structure. The process involves selective acid hydrolysis of solvent accessible and amorphous regions of secondary structure in refined cellulose to produce relatively crystalline microdomains that are resistant to further hydrolysis. The dimension of the crystallite domains is on the order of ten to thirty microns. If the never dried crystallite is sheared, it disperses into parallel clusters of microfibrils, reflecting periodic cleavage along a fibril assembly. The microfibril crystallites exhibit high surface area and readily reassociate on drying into a hard, non-dispersible mass.

Furthermore, the production of rayon and cellulose ethers such as cellulose gum (carboxymethyl cellulose, CMC) involves manipulation at the primary level of structure. In the case of rayon the modification is transient and reversible whereby the reconstituted beta-glucan chain spontaneously reassembles into semi-crystalline material that can be spun into fibrils. Cellulose ethers represent a deliberate, irreversible modification whereby the individually formed beta-glucan chains are prevented from reassembly due to the chemical derivatization. A limited variation of such derivatization is that of powdered cellulose wherein the degree of substitution is relatively low, to form e.g. forming carboxymethyl or diethyl aminoethyl cellulose, CM cellulose and DEAE cellulose, respectively. The latter materials are useful as ion exchange media.

SUMMARY OF THE INVENTION

It is an object of this invention to provide a relatively simple and inexpensive means for refining fibrous cellulosic material into a dispersed tertiary level of structure and thereby achieve the desirable properties attendant with such structural change. The cellulosic fiber produced in this way is referred to herein as "microdenominated cellulose (MDC)".

The foregoing object is achieved by repeatedly passing a liquid suspension of fibrous cellulose through a zone of high shear, which is defined by two opposed surfaces, with one of the surfaces rotating relative to the other, under conditions and for a length of time sufficient to render the suspension substantially stable and to impart to the suspension a Canadian Standard Freeness that shows consistent increase with repeated passage of the cellulose suspension through the zone of high shear.

It has now been discovered that microdenominated cellulose can be produced using standard refining equipment, e.g. a double disk refiner, operated in a way differing from the conventional use of this equipment in refining pulp for paper manufacture. Whereas paper manufacture calls for minimum damage to the fiber during refining and a Canadian Standard Freeness consistent with good drainage of water from the pulp, it will be apparent from the following disclosure that use of the same equipment may be employed to achieve the opposite effect, i.e., a high degree of disintegration of the fiber structure, which results in a cellulose product having very high surface area and high water absorbency. The degree of disintegration is sufficiently severe that, as refining continues beyond that level normally used for paper manufacture (a Canadian Standard Freeness value approximating 100), a reversal of the Canadian Standard Freeness values occurs. The reason for this reversal is that the dispersed fiber becomes sufficiently microdenominated that gradually greater amounts of fiber begin to pass through the perforated plate of the Canadian Standard Freeness tester with water, thus leading to a progressive increase in the measured value as refining continues. Continuation of refining ultimately results in essentially all of the refined fiber readily passing through the perforated plate with water. At this stage of processing, the measured Canadian Standard Freeness value is typical of that for unimpeded passage of water through the perforated plate of the test unit.

Whereas a single stage, and at most two stages are used for conventional refiner processing in paper manufacture, the process of this invention requires multiple passages of the pulp through the zone of high shear, which may typically involve ten to forty passages.

In paper manufacture beating or refining increases the area of contact between dispersed fibers by increasing the surface area through dispersion and fibrillation. MDC manufacture applies and extends such processing to a much greater degree. It is believed that the extent of refinement needed to achieve this high degree of fibrillation leads to a concomitant disassembly of tertiary structure, and perhaps even secondary structure. The result is an ultrastructurally dispersed form of cellulose with very high surface area.

The process for preparing MDC is more closely associated with disassembly than it is with the indiscriminate fragmentation used in mechanical comminution or grinding of dried, refined cellulose or micromilling of cellulose in liquid media. It is also quite different from the approaches noted above based on chemical hydrolysis or chemical modification. The product, MDC, is most nearly like microfibrillated cellulose, MFC, produced by high pressure impact discharge of a cellulosic

dispersion in a liquid medium onto a solid surface, as disclosed by Turbek et al. However, contrary to the teachings in the Turbek et al patent, to the effect that beating and refining as practiced in the paper industry are relatively inefficient processes since large amounts of energy are expended to gain relatively minor amounts of fiber opening and fibrillation, the opposite appears to be true based on the research leading up to this invention, as will be explained below.

The MDC product of the invention has very high surface area, consisting essentially of thread-like structures (most of which are not discernable with the light microscope). These represent longitudinally oriented clusters of microfibrils with attendant, protuberant ultrastructure emanating from their surfaces. These structures form entangling and interacting networks which lead to a unique form of microscopic compartmentalization for mixtures of discontinuous materials in water or other continuous phase systems. Such behavior results in the formation of interesting viscoelastic characteristics such as gel structure, mouthfeel, textural quality and other properties highly desired in foods, pharmaceutical and cosmetics products.

The product of the invention, MDC, is characterized by a settled volume greater than about 50% after twenty-four hours, as based on 1% by weight aqueous suspension, and water retention greater than about 350%. Procedures for determining the settled volume and water retention values for MDC are described in detail below.

BRIEF DESCRIPTION OF THE DRAWINGS

FIG. 1 is a photomicrograph of wheat fiber described in EXAMPLE 1 below before refining shown at a magnification of 100 times.

FIG. 2 is a photomicrograph of the aforesaid wheat fiber after refining shown at a magnification of 100 times.

FIG. 3 is a photomicrograph of the aforesaid wheat fiber before refining shown at a magnification of 250 times.

FIG. 4 is a photomicrograph of the aforesaid wheat fiber after refining shown at a magnification of 250 times.

FIG. 5 is a photomicrograph of softwood fiber described in EXAMPLE 2 below before refining shown at a magnification of 100 times.

FIG. 6 is a photomicrograph of the aforesaid softwood fiber after refining shown at a magnification of 100 times.

FIG. 7 is a photomicrograph of the aforesaid softwood fiber before refining shown at a magnification of 250 times.

FIG. 8 is a photomicrograph of the aforesaid softwood fiber after refining shown at a magnification of 250 times.

FIG. 9 is a photomicrograph of oat fiber described in EXAMPLE 3 below before refining shown at a magnification of 100 times.

FIG. 10 is a photomicrograph of the aforesaid oat fiber after refining shown at a magnification of 100 times.

FIG. 11 is a photomicrograph of the aforesaid oat fiber before refining shown at a magnification of 250 times.

FIG. 12 is a photomicrograph of the aforesaid oat fiber after refining shown at a magnification of 250 times.

DETAILED DESCRIPTION OF THE INVENTION

In accordance with the present invention, microdenominated cellulose is produced from cellulosic material by repeatedly passing the material in an aqueous suspension through a zone of high shear, defined by two opposed surfaces, one of which is caused to rotate relative to the other. According to a preferred embodiment, the cellulose suspension is passed through a double disk refiner of the type typically used in the processing of wood pulps for paper manufacture. Whereas processing with such equipment in conventional paper applications is limited so that the degree of refining achieved corresponds to Canadian Standard Freeness (CSF) values of about 100 or greater, the present invention calls for a degree of refinement whereby the CSF value is reduced toward zero and then progresses through freeness values, approaching and ultimately exceeding the values for never-processed pulp in aqueous suspension.

Examples of the use of this invention are set forth below for softwood pulp, white wheat fiber and oat fiber. Other types of cellulosic fibrous material can also be processed in accordance with the present invention. However, long-fibered materials such as the softwood pulp and wheat fiber appear to be better suited to this approach than short-fibered material.

The starting material for the process is conveniently prepared by beating cellulosic sheet material in a hydrobeater in the presence of a suitable liquid, which disintegrates the sheet material and uniformly disperses the fibers in the liquid.

The exact amount of refining time required to produce MDC depends on the characteristics of the starting material e.g. the fiber length, the temperature of refining and the solids concentration in the pulp. The length of processing is also influenced by the parameters of the shear zone in which the cellulose suspension is processed. In the case of a double disk refiner, these parameters include the amount of back pressure exerted on the cellulose suspension as it is subjected to shear stress during refining, the refiner plate surface configuration, the space between confronting refiner plates, refiner plate diameter and plate peripheral speed. Efficiency is enhanced by operation at high pulp solids concentration, an elevated back pressure on the pulp during refining, elevated pulp temperatures coupled with maximum temperature control, adjustment of the gap between confronting refiner plates by keying on a pre-selected value of amperage to the refiner motor and a refiner plate configuration and peripheral speed that promotes "rubbing" or fraying rather than cutting. Although refining proceeds most efficiently as the solids concentration in the pulp is increased, however, there is a limit to how high the solids concentration can be and still have the pulp flow through the system. A short-fibered material like oat can be concentrated to almost twice the solids concentration possible with softwood and wheat, both long-fibered materials.

Preferred operating conditions for preparation of MDC in a double disk refiner are as follows: fiber length of about 50 to 3000 microns, or greater; refining temperature of about 60° F. to about 200° F.; a solids concentration of about 2 to about 10% by weight of the cellulose suspension; and back pressure of about 10 to about 40 psi.

The remaining parameters, including plate configurations, spacing between adjacent plates, plate diameter and peripheral plate speed will depend on the particular model of refiner selected to process the MDC. A typical run employing a Black Clawson 28-inch Twin Hydradisc refiner is exemplified below.

A primary indicator used to monitor the extent of refining of the cellulosic material is the Canadian Standard Freeness value as measured using test equipment and procedures contained in TAPPI 227 "Freeness of Pulp" J. Casey, *Pulp and Paper* (1980). Freeness has been shown to be related to the surface conditions and the swelling of fiber which influences drainage. As refining continues beyond levels normally practiced in conventional paper making, the dimensions of the resulting structures become sufficiently small such that a reversal of freeness values occurs, i.e. increasing rather than diminishing values of freeness as refining continues. This anomalous rise of freeness is referred to herein as "false freeness". Once the reversal occurs and refining continues thereafter, the measured freeness value increases until a maximum value of approximately 800 is reached. At this point the refined material has been rendered sufficiently supple and fine (dimensionally small) that it readily passes through the perforations of the perforated plate of the tester along with the water. In other words, the suspension behaves as though it were fiber-free water of the same total volume as the fiber-containing sample being measured. This is the limiting condition for obtaining meaningful data from freeness measurements. As the cellulose suspension achieves this desired level of freeness, it becomes substantially stable, which is intended to mean that there is no visible segregation of the continuous phase from the disperse phase, even upon standing for a reasonable period of time.

Examination of photomicrographs of fiber samples provide insight as to the degree of fibrillation that is achieved by refining, with reference to the starting material. FIG. 1 shows that the length of the fiber prior to refining is in most cases at least 1000 microns and the fiber width is one to two microns. Shown in FIG. 2 is the wheat fiber structure resulting from the refining process described in EXAMPLE 1, below, at a magnification of 100 times. FIG. 3 and FIG. 4 show the wheat fiber at a magnification of 250 times and reveal detail regarding the refined fiber in FIG. 4. It is apparent from FIG. 2 and FIG. 4 that the refined fiber is highly disassembled. There is no evidence of the original quaternary structure shown in the fiber before refining. It has been disintegrated by the extended period of refining and replaced by a network of fibrils of vastly increased surface area. These fibrils as viewed in the light microscope, appear as very long threadlike strands of extremely small diameter for those that can be seen.

FIG. 5 shows the fiber structure of softwood fiber at a magnification of 100 times before refining and reveals a somewhat longer length (1000 to 3000 microns long) and greater width (two to four microns wide) than the wheat fiber described above. FIG. 6 and FIG. 8 show fiber structure for the refined softwood that appears to be quite similar to that of the wheat fiber sample discussed above.

Examination of photomicrographs of oat fiber samples provide insight as to the influence of fiber length of the starting material on the degree of fibrillation achieved by refining as compared to the longer fiber starting materials. FIG. 9 shows the oat fiber prior to refining to be between 500 and 1000 microns in length and two to four microns in width. FIG. 10 and FIG. 12 show that refined oat fiber structure undergoes disassembly but not to the degree of the long fibered wheat and softwood samples. There is some evidence of the original quaternary structure shown in the fiber before refining. A smaller percentage of the structure of oat fiber has been converted to a network of fibrils. This has resulted in less surface area being created than occurs when long fibered materials are refined.

As will be appreciated from the foregoing description, MDC is the result of disassembly of cellulose structure via essentially physical manipulation. As such, MDC is distinguishable from cellulosic products produced by chemical transformation. No appreciable chemical change of the cellulose starting material occurs during the refining process described herein.

Several other parameters or properties, in addition to Canadian Standard Freeness, serve to characterize MDC.

A parameter useful in the characterization and description of MDC is the settled volume of aqueous dispersions of differing solids content after twenty-four hours of settling. The settled volume of a sample of MDC is determined by dispersing a known weight of cellulose (dry weight basis) in a known amount of water, e.g. in a graduated cylinder. After a prescribed settling time, the volume of the bed of suspended cellulose is measured with reference to the total volume of the continuous aqueous phase. The settled volume is expressed as a percentage of the bed volume to the total volume. From this data the solids concentration in an aqueous dispersion that results in a settled volume that is fifty percent of the original volume can be determined and used to characterize the product. The results of such measurements are shown in Table 1. Ultrastructural parameters are also important in this characterization. The very long fibril softwood has the lowest solids concentration for 50% settled volume at 0.18%. The intermediate wheat fiber is next at 0.23% and oat fiber with a very short fibril is highest at 0.87%. A characteristic of MDC is that a 1% by weight aqueous suspension has a settled volume greater than 50% after twenty-four hours.

TABLE 1

Example Number	Fibrous Material	False Value of CSF (ml)	Viscosity at 1.5 Wt. % (cp)	50% Settled Volume Wt.	% Water % Retention
1	Wheat	780	5,860	0.23	1,005
2	Softwood	730	7,850	0.18	1,110
3	Oat	810	1,300	0.76	569

Water retention is another parameter for characterizing MDC. Water retention values are determined by employing a pressure filtration apparatus (Baroid Model 301 for low pressure fluid loss control measurements, N. L. Baroid Corporation, Houston, Tex.) routinely used to evaluate drilling fluid properties. A 100 gram aliquot of a nominal 4 to 8% w/w aqueous dispersion of cellulose is loaded into the filter cell chamber, the cell chamber is capped and subjected to 30 psig. pressure from a regulated nitrogen source. The water discharged from the filtration cell chamber is collected and pressure continued for thirty seconds after observation of the first gas discharge. The nitrogen source is then turned off and collection of discharged water con-

tinued for one minute or until the gas discharge ceases, whichever event occurs first. Basically the technique employs pneumatic, pressure filtration to remove interstitial water from the particulate phase.

The expressed volume of water is recorded along with the weight of wet cake. The wet cake is then dried for sixteen hours at 95 degrees Centigrade or until a constant weight is recorded. The water retention value is computed as the ratio of (wet cake weight minus the dry cake weight) to (dry cake weight) times 100. This technique provides a good estimate of the capillary and absorptive retention of water by the cellulose solids by removing the interstitial water from the cake solids. The procedure is quick (5 to 10 minutes) and highly reproducible. The water retention value of MDC is characteristically at least 350%, and preferably at least 500%.

Viscosity may also be used as a characterizing property of MDC. Apparent viscosities of an aqueous dispersions of 1.5% w/w MDC solids samples were determined with a Brookfield Viscometer model DV-III using spindle SC4-16 with the small cell adapter at a number of shear conditions (5 through 100 RPM). The samples were pre-dispersed by high speed mixing for three minutes at 10,000 RPM with a rotor stator type mixer (Omni International, model 1000). The viscosities measured for final refined product (MDC) of the three examples are shown in Table 1. The softwood fiber product exhibited a viscosity of approximately 8,000 centipoise at a spindle speed of 100 RPM. The white wheat fiber product had a viscosity of approximately 6,000 and the oat fiber a viscosity of approximately 1,300 at the same measurement conditions as for the softwood fiber. It appears the wide range in the measured viscosities is primarily due to the differences in fibril length and other ultrastructural characteristics of the starting materials.

It should be understood that the above viscosity measurements on MDC dispersions are made on a heterogeneous mixture (an interacting particle ensemble suspended in a fluid medium). Viscosity measurement is normally applied to homogenous systems. Because of the heterogeneous nature of the mixture a certain degree of mechanical distortion occurs in the mixture around the rotating spindle used to determine shear stress forces within the mixture. Consequently shear/shear stress measurements are time and history dependent. As such the measurement is not a true viscosity in the conventional sense but rather provides a reproducible measurement that has been found useful for characterizing the degree of microdenomination and in describing the implementation of this invention.

Energy input for refining MDC in the manner described herein ranges from about 0.5 to about 2.5 kilowatt-hours per pound of MDC (dry weight basis) and associated refining times vary from two to eight hours depending on the cellulosic material being processed. This is significantly lower than the energy requirements for microfibrillated cellulose as reported by Turbak et al in U.S. Pat. No. 4,483,743. Based on five to ten passes of a 1% MFC solids aqueous dispersion through an 80% efficient homogenizer at 8,000 psig. the energy requirement ranges from 4.4 to 8.7 kilowatt-hours per pound of MFC.

The following examples are provided to describe in further detail the preparation of MDC in accordance with the present invention. These examples are intended to illustrate and not to limit the invention.

EXAMPLE 1

Never dried white wheat fiber was mixed with 2,190 gallons of water in a hydrobeater (Black Clawson Model 4-SD-4 with Driver No. 45) to make up a pulp of 4.5% w/w solids. The white wheat fiber used in this example is a commercially available refined fiber product derived from bleached wheat chaff obtained from Watson Foods Company, West Haven, Conn. The white wheat product was obtained as a nominal 40% w/w nonvolatile solids fiber mat. The product was stated to be 98% total dietary fiber by the Prosky method. The particle size by microscopic examination indicated a largely heterogeneous population of thin needle-like slerchyma cells ranging in major/minor dimensions of 500 to 1000 / 10 to 20 microns with few interspersed parenchyma cells of 200/50 microns.

After beating the pulp for twenty minutes at room temperature it was transferred to a water jacketed holding tank to be repeatedly passed through a Black Clawson Twin Hydradisc refiner. The refiner of this example is a twenty-eight inch diameter double disc unit powered by a 250 horsepower motor. The refiner plates mounted on the discs are made of sharloy (a nickel hardened steel). The refiner plates were not equipped with dams. The faces of the particular refiner plates used in this refiner consists of alternate bars and grooves oriented so that bars of the adjacent refiner plates (one static and the other revolving) move relative to one another with a scissoring action occurring as the bars of each confronting plate move past one another. The three critical dimensions of these bars and grooves are the bar width, channel width and channel depth. For this particular unit, they were, respectively, 2/16 of an inch, 4/16 of an inch and 3/16 of an inch (expressed as 2,4,3 by Black Clawson's convention).

The refiner plates on the revolving disc move at 713 revolutions per minute. Based on the outer periphery of the refiner disc extending to 13 and ¼ inch from the centerline of the drive shaft, this corresponds to peripheral speed of about 4,900 feet per minute. The pulp was continuously circulated at a rate of approximately 250 gallons per minute through the refiner and back to the holding tank. Passage of the cellulose suspension through the refiner occurs so as to have equal flow on each side of the revolving disc.

One disc of the refiner is fixed while the other is sliding. This allows the distance between adjacent discs to be adjusted. In the full open position (typical of startup or shutdown), discs are one and three-quarters inch apart. During refining, the discs are of the order of one to two thousands of an inch apart. Rather than adjust the gap between discs to a specific spacing, the value of the amperage to the motor driving the refiner is used to establish spacing. The procedure upon startup is to move the discs from the full open position to a closer position where the amperage reading increases until it reaches 310 amps. At this point, maximum power is being delivered from the motor. Once this point is reached, the back pressure on the refiner is increased by closing down the valve on the line returning pulp from the refiner to the holding tank. The back pressure is normally raised from an initial value of about 14 psig to a final value of about 35 psig. As the back pressure is increased without adjustment of the sliding disc location, the amperage drawn by the motor decreases to about 260 amps. With the back pressure at 35 psig, the sliding disc is adjusted to bring the discs closer together

until the desired 310 amps are drawn by the motor. Once this is done, there is no further adjustment of the sliding disc unless the motor amperage drops significantly. This may occur as refining proceeds if certain properties of the pulp change significantly. In that event, the sliding disc is moved to reduce the gap between the discs until either the desired amperage is once again achieved, or the discs begin to squeal. Squealing is to be avoided as it is indicative of excessive disc wear and leads to high refiner plate replacement costs.

A gate-type mixer in the holding tank continuously mixed the contents during refining. A back pressure of 34 pounds per square inch was maintained in the return line from the refiner outlet to the holding tank. The recycle operation continued for approximately six hours during which the Canadian Standard Freeness of the pulp changed from an initial value of 190 to a final "false" value of 780 ml.

During refining the temperature of the pulp increased from an initial value of 64 to a final value of 190 degrees Fahrenheit. The amperage drawn by the 250 horsepower motor of the refiner varied from 310 initially to 290 amperes at completion of refining. Energy input to the refiner was approximately 1.2 kilowatt-hours per pound of refined fiber processed (dry weight basis). The resulting product is characterized in TABLE 1.

EXAMPLE 2

Dry, softwood fiber used in this example was obtained from Stora Forest Industries Ltd., Port Hawkesbury, Nova Scotia, Canada as a bleached sulfite pulp. It was derived from softwood species (balsam fir and black and white spruce). The bleaching sequence was reported to be (D70+D70) E (DE) D. The ash (TAPPI 211 and 85) is 0.6% and the CSF 660. The dispersed individual fibers appeared to be 20 to 25 microns in diameter and ranged from one to three mm. in length with the average fiber 25 microns by 2 mm.

Sheets of dry, softwood fiber (Storafite 04-620972) were mixed with 2,080 gallons of water in the same hydrobeater as used in EXAMPLE 1 to make up a pulp of 3.7% solids. After beating the pulp for twenty minutes at room temperature it was transferred to the holding tank to be repeatedly passed through the same Black Clawson refiner as used in EXAMPLE 1. Pulp was circulated at a rate of approximately 250 gallons per minute through the refiner and back to the holding tank. A gate-type mixer continuously mixed the contents of the holding tank during refining. A back pressure of 34 pounds per square inch was maintained in the return line from the refiner outlet to the holding tank. The recycle operation continued for approximately six hours during which the Canadian Standard Freeness of the pulp changed from an initial value of 620 to a final "false" value of 730 ml.

During refining the temperature of the pulp increased from an initial value of 64 to a final value of 144 degrees Fahrenheit. The amperage drawn by the 250 horsepower motor of the refiner varied from 310 initially to 290 amperes at completion of refining. Energy input to the refiner was approximately 2.4 kilowatt-hours per pound of refined fiber processed (dry weight basis).

EXAMPLE 3

Dry oat fiber (Williamson Type 9780) was mixed with 1,055 gallons of water directly into the holding tank for the refiner to make up a pulp of 7.86% solids. The dry oat fiber used in this example is a commercially available refined fiber product derived from bleached oat hulls (from Opta Food Ingredients, Inc. in Cambridge Mass.). The product, identified as Better Basics TM type 780, is stated to be 98% total dietary fiber by the Prosky method. It was obtained as a dry, light tan colored powder that was readily hydrated in the refiner tank prior to refining. The particle size was such that 98% on a weight basis passed through a 50 mesh screen using an Alpine Airjet Sieve. Microscopic examination indicated particles consisted largely of heterogeneous dispersed fiber cells with major/minor dimensions of 100 to 600 / 10 to 40 microns. In contrast to wheat and softwood oat represents a relatively short fiber structure.

The already finely divided state of the oat fiber made it possible to eliminate the hydrobeater step. The pulp was refined in the same Black Clawson unit as used in the two previous examples. Pulp was circulated at a rate of approximately 250 gallons per minute through the refiner and back to the holding tank. A gate-type mixer in the holding tank continuously mixed the contents during refining. Back pressure maintained in the return line from the refiner outlet to the holding tank varied from 34 to 31 pounds per square inch gauge. The recycle operation continued for two hours and forty minutes during which the Canadian Standard Freeness of the pulp changed from an initial value of 310 to a final "false" value of 810 ml.

During refining the temperature of the pulp increased from an initial value of 65 to a final value of 168 degrees Fahrenheit. The amperage drawn by the 250 horsepower motor of the refiner varied from 310 initially to 260 amperes at completion of refining. Energy input to the refiner was approximately 0.5 kilowatt-hours per pound of refined fiber processed (dry weight basis).

While certain preferred embodiments of the present invention have been described and examplified above, it is not intended to limit the invention to such embodiments, but various modifications may be made thereto, without departing from the scope and spirit of the present invention as set forth in the following claims.

We claim

1. A process for preparing microdenominated cellulose comprising repeatedly passing a liquid suspension of fibrous cellulose through a zone of high shear, said zone being defined by two confronting refining disk surfaces, with one of said surfaces rotating relative to the other, under conditions and for a length of time sufficient to render said suspension substantially stable and to impart to said suspension a Canadian Standard Freeness that shows consistent increase with repeated passage of said cellulose through said zone of high shear.

2. A process as claimed in claim 1 wherein said suspension is passed through a zone of high shear defined by confronting disk surfaces of a double disk refiner.

3. A process as claimed in claim 1 wherein said suspension contains 2 to 10% by weight of cellulose.

4. A process as claimed in claim 1 wherein said suspension is an aqueous suspension.

5. A process as claimed in claim 1 wherein said liquid suspension is maintained at an elevated temperature no greater than 200° F.

6. A process as claimed in claim 1 wherein a back pressure of at least 30 psig is exerted on said liquid suspension in said zone of high shear.

7. A process as claimed in claim 1 wherein said liquid suspension of fibrous cellulose material is prepared by beating sheets of cellulose in a hydrobreater in the presence of said liquid.

* * * * *

X. RELATED PROCEEDINGS APPENDIX

NONE